

**Submission ID #: 70205**

**Scriptwriter Name: Poornima G**

**Project Page Link: <https://review.jove.com/account/file-uploader?src=21412573>**

**Title: Piezoreflectance Spectroscopy of Optical Transitions in van der Waals Layered Crystals**

**Authors and Affiliations:**

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## **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? **Yes, all done!**
- 3. Filming location:** Will the filming need to take place in multiple locations? **No**
- 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: 25

Number of Shots: 59 (11 SC; 3 added shots)

# Introduction

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

**Authors:** Please note that **the questions will not appear on screen**. Please answer in **stand-alone full sentence with sufficient context**

## INTRODUCTION:

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

- 1.1. **Katarzyna Hołodnik-Małecka:** My research develops piezoreflectance spectroscopy to identify direct optical transitions in layered semiconductors and understand strain-driven spectral changes.

1.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera

~~Describe the research contexts or systems in which this protocol can be applied.~~

- 1.2. **Katarzyna Hołodnik-Małecka:** This method is suitable for micrometer-scale optical studies of layered semiconductors, including bulk and few-layer van der Waals crystals.

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. **Katarzyna Hołodnik-Małecka:** It allows researchers to detect strain-responsive direct optical transitions in those materials with enhanced sensitivity.

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. **Katarzyna Hołodnik-Małecka:** The key challenge is maximizing signal-to-noise ratio while ensuring efficient strain transfer and stable sample mounting for reliable optical measurements.

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

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

1.5. **Katarzyna Hołodnik-Małecka:** Future studies can extend this method to monolayers and heterostructures to explore strain effects and optical transitions in more complex systems.

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.*

NOTE: SCREEN timestamps for protocol were added at the postshoot stage. Please contact the postshoot note integrator (Kathyayani Sridharan) for queries regarding SCREEN shots.

## 2. Preparation and Transfer of van der Waals Crystal Flakes onto Piezoceramic Substrate

~~Preparation and Transfer of Exfoliated van der Waals Crystal Flakes onto the Piezoceramic Substrate~~

**Demonstrator:** Katarzyna Hołodnik-Matecka

2.1. To begin, obtain the bulk material of a van der Waals crystal such as Tungsten Disulfide, or  $WS_2$  (*W-S-two*) for optical characterization [1]. Thin the material by repeatedly cleaving it using adhesive tape [2] until the preferred thickness is reached [3].

2.1.1. WIDE: Talent placing the crystal on the work surface.

2.1.2. Talent repeatedly peeling adhesive tape to cleave a bulk crystal and reduce its thickness.

Added shot: Close-up of van der Waals crystal with preferred final thickness.

**Videographer's notes:** Additional shot 2.1.2.A to be placed right after 2.1.2 (this is the CU)

2.2. Transfer the thinned fragments from the tape to a polydimethylsiloxane or PDMS (*P-D-M-S*) stamp by placing the stamp onto the tape [1]. After approximately five minutes, peel back the PDMS [2] so that flakes retained on the PDMS can be used for subsequent transfer [3].

2.2.1. Talent laminating the PDMS stamp onto the adhesive tape containing thinned fragments.

2.2.2. Talent peeling back the PDMS stamp ~~with flakes remaining on its surface.~~

Added shot: Close-up of crystal flakes on PDMS surface.

**Videographer's notes:** Additional shot 2.2.2.A to be placed right after 2.2.2 (this is the CU)

2.3. Clean the piezoceramic surface by rinsing it with acetone [1] and dry the surface using clean, dry air or nitrogen [2].

2.3.1. Talent rinsing the piezoceramic surface with acetone.

2.3.2. Talent drying the piezoceramic surface with a stream of clean, dry air or nitrogen.

- 2.4. Now, apply a very thin layer of an adhesive medium to the piezoceramic [1].
  - 2.4.1. Talent applying a thin layer of adhesive medium onto the piezoceramic surface.
- 2.5. Immediately place the PDMS carrying the flake onto the wet adhesive on the piezoceramic [1]. Press gently to ensure contact [2]. Wait approximately thirty seconds and peel off the PDMS so that the material remains on the piezoceramic [3]. Verify adhesion by visual inspection or confirm under an optical microscope if needed [4].
  - 2.5.1. Talent placing the PDMS stamp with the flake onto the adhesive-coated piezoceramic.
  - 2.5.2. Talent gently pressing the stamp to ensure contact with the surface.
  - 2.5.3. Talent peeling off the PDMS stamp, leaving the material on the piezoceramic.
  - 2.5.4. Shot of the transferred material on the piezoceramic surface.

**Note:** Shots 2.4.1, 2.5.1, 2.5.2, 2.5.3 are filmed as one shot. 2.5.4 filmed as 2.5.2.A.

**Videographer's note:** Shot according to the slate as 2.5.2.A should be put in place of 2.5.4.

### **3. Electrical Contact Fabrication and System Integration of Piezoceramic Sample** ~~**Fabrication of Electrical Contacts and Integration of the Piezoceramic Sample into the Experimental Setup**~~

- 3.1. Apply a silver paste to secure the piezoceramic [1] and place the prepared piezoceramic containing the transferred sample on the stage to ensure electrical contact with the bottom electrodes of the piezoceramic [2].
  - 3.1.1. Talent applying silver paste to secure the piezoceramic and establish electrical contact with the bottom electrodes.
  - 3.1.2. Talent placing the piezoceramic with the transferred sample onto the sample stage.
- 3.2. Form the top electrical contact using silver paste and copper wire [1]. After applying the silver paste, allow it to dry and cure at room temperature for approximately ten hours before proceeding [2].
  - 3.2.1. Talent applying silver paste and attaching a copper wire to create the top electrical contact.
  - 3.2.2. Talent placing the sample aside for curing.

3.3. Then, insert the mounted sample into the optical setup [1]. Connect the electrodes to the alternating current or the AC (A-C) voltage generator and to the ground using insulated wires [2]. Keep all exposed conductors insulated and positioned away from the optical path [3].

3.3.1. Talent placing the mounted sample into the optical setup.

3.3.2. Talent connecting insulated wires from the electrodes to the AC voltage generator and ground.

3.3.3. Talent arranging insulated conductors away from the optical path.

#### **4. AC Reflectance Component ( $\Delta R$ ) Measurement**

##### **Measurement of AC Reflectance Component ( $\Delta R$ ) Using Lock-In Detection**

4.1. Turn on the halogen light source and set it to maximum output [1]. Ensure that the optical chopper [2] is not obstructing the monochromator entrance slit [3].

4.1.1. Talent switching on the halogen light source and adjusting it to maximum output.

4.1.2. Talent checking and showing ~~that the optical chopper is clear of the monochromator entrance slit.~~

Added shot: Close-up of optical chopper clear of monochromator entrance slit.

**Videographer's notes:** Additional shot 4.1.2.A to be placed right after 4.1.2

4.2. Route the beam to the charge-coupled device camera [1] and align the spot on the selected region of the sample using the XYZ microcontrollers [2]. Then, adjust the iris diaphragm aperture to set the spot diameter on the sample [3].

4.2.1. Talent directing the beam path toward the charge-coupled device camera.

4.2.2. Talent adjusting XYZ microcontrollers to align the light spot on the sample.

4.2.3. Talent adjusting the iris diaphragm to control the spot size on the sample.

4.3. Switch the signal path from the charge-coupled device preview to the silicon PIN photodiode detector by removing beamsplitter from optical path [1].

4.3.1. Talent switching the signal path from the charge-coupled device preview to the silicon PIN photodiode detector.

4.4. Next, configure the preamplifier by setting the bias voltage, filter type and cutoff, input

offset, gain mode, and sensitivity to achieve a stable signal without saturation [1]. Document the selected values [2].

4.4.1. Talent adjusting preamplifier controls to set bias voltage, filter type, cutoff, input offset, gain mode, and sensitivity.

4.4.2. Talent taking a picture of the settings.

4.5. Configure the lock-in amplifier to display the in-phase X component [1]. Set the reference frequency for AC voltage generator and select the internal reference source [2]. Then, choose an appropriate time constant and sensitivity [3].

4.5.1. Talent setting the lock-in amplifier display to the X in-phase component.

4.5.2. Talent adjusting the reference frequency and selecting the internal reference source.

4.5.3. Talent setting the time constant and sensitivity on the lock-in amplifier.

4.6. Now, enable the AC voltage generator and set the desired amplitude [1]. Verify that the signal reaches the piezoceramic without overdriving the device [2].

4.6.1. Talent turning on the AC voltage generator and adjusting the voltage amplitude.

4.6.2. Talent checking the signal delivery to the piezoceramic to avoid overdriving.

4.7. Launch the **LabVIEW 2018** (*LAB-View-Two thousand eighteen*) application and select the option **OSN23.vi** (*O-S-N-twenty-three-dot-V-I*) [1]. Within the measurement parameters section, set the measurement mode as **XY (X-Y)** [2]. Set the **Pre-delay**, **Averaging** and **Delay** parameter values [3] and specify the output text file path for saving the data [4].

4.7.1. SCREEN: 471. 00:00:00 – 00:00:04

4.7.2. SCREEN: 471. 00:00:05 – 00:00:17

4.7.3. SCREEN: 472. 00:00:00 – 00:00:07

4.7.4. SCREEN: 472. 00:00:08 – 00:00:15

4.8. Next, define the **Start** and **Stop** values of photon energy [1] and set the monochromator **Step** value according to instrument limits [2]. After setting the values, start the scan, initialize the devices, then start measurement [3] to record the progress over a range of energy and wavelength values [4].

4.8.1. SCREEN: 473. 00:00:00– 00:00:05

4.8.2. SCREEN: 473. 00:00:06– 00:00:08

4.8.3. SCREEN: 481. 00:00:00– 00:00:11

4.8.4. SCREEN: 482. 00:00:00– 00:00:24

4.9. After completion, turn off the AC voltage generator without disturbing the sample position [3].

4.9.1. Talent switching off the AC voltage generator, leaving the sample undisturbed.

## **5. DC Reflectance Component (R) Measurement**

### **~~Measurement of DC Reflectance Component (R) using Chopper Referenced Lock-In Mode~~**

5.1. Confirm that the AC voltage generator remains off [1].

5.1.1. Talent verifying and showing that the AC voltage generator is switched off.

5.2. Keep the beam position on the sample without alteration [1]. To ensure the focus is maintained, route the beam to the charge-coupled device preview [2], refocus and re-center the spot [3], then return the signal path to the photodiode [4].

5.2.1. Talent pointing to the beam position fixed on the sample.

5.2.2. Talent switching the beam path to the charge-coupled device preview.

5.2.3. Talent adjusting focus and re-centering the beam spot on the sample.

5.2.4. Talent switching the signal path back to the photodiode detector.

5.3. Start the optical chopper using its controller and set the frequency equal to the previously used modulation frequency [1]. Verify that the chopper reference output is connected to the lock-in reference input [2].

5.3.1. Talent operating the optical chopper controller to start the chopper and set the frequency.

5.3.2. Talent checking the connection between the chopper reference output and the lock-in reference input.

5.4. Keep the preamplifier settings unchanged. Configure the lock-in amplifier to display the referenced reflectance R and select the chopper controller as the external reference source [1]. Set input parameters identical to the previous measurement and adjust the time constant and sensitivity to maintain a stable signal [2].

- 5.4.1. Talent setting the lock-in amplifier to R display and selecting the external reference source from the chopper controller.
- 5.4.2. Talent adjusting time constant and sensitivity while keeping other parameters consistent.
- 5.5. In the **LabVIEW 2018** application within the measurement parameters section, update the measurement mode as **R-phase (R-face)** [1] Update the **Pre-delay** value [2] and specify a new output text file path to save the data [3]. ~~Name the files to encode the sample, location, and acquisition parameters for pairing datasets [3].~~
- 5.5.1. SCREEN: 551. 00:00:00– 00:00:08
- 5.5.2. SCREEN: 552\_553: 00:00:00– 00:00:04
- 5.5.3. SCREEN: 552\_553: 00:00:05– 00:00:17
- Note:** Shots 5.5.2 and 5.5.3 were combined into one screenshot. As per videographer's note, shots no. 5.5.2+ 5.5.3 recorded together, in one take.
- 5.6. Now, set the same photon energy values for Start, Stop and Step parameters as previously demonstrated [1]. Start the scan and allow it to run to completion [2].
- 5.6.1. SCREEN: 561. 00:00:00– 00:00:09
- 5.6.2. SCREEN: 562. 00:00:00– 00:00:24
- 5.7. After completion, turn off the optical chopper [1]. If no further measurements are planned, turn off the light source, monochromator, and other instruments [2].
- 5.7.1. Talent switching off the optical chopper using the controller.
- 5.7.2. Talent powering down the light source, monochromator, and associated instruments.
- 5.8. After labelling the datasets, compute and plot the normalized  $\Delta R/R$  (*delta R by R*) spectrum versus photon energy to identify optical transitions [1]. Finally, fit the spectrum using the Aspnes line-shape model [2] by selecting energy range including resonance, refining parameters iteratively, and validating the fit based on residuals and stability [3].
- 5.8.1. SCREEN: 581. 00:00:00– 00:00:09
- 5.8.2. TEXT ON PLAIN BACKGROUND:
- $$\frac{\Delta R}{R}(E) = Re \left[ \sum_{j=1}^n C_j e^{i\theta_j} (E - E_j - i\Gamma_j)^{-m_j} \right] + y_0 + aE + bE^2$$
- 5.8.3. SCREEN: 583. 00:00:00– 00:00:19

**Videographer's Note:** Shot numbers. 5.8.1 and 5.8.3 (screen as a backup) were not recorded because the author did not prepare them.

# Results

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## 6. Results

6.1. The strain-modulated AC component  $\Delta R$  (*delta R*) [1] and the DC component proportional to reflectance  $R$  were measured for a single spot in tungsten disulfide [2].

6.1.1. LAB MEDIA: Figure 6A. *Video editor: Highlight the red  $\Delta R$  trace*

6.1.2. LAB MEDIA: Figure 6A. *Video editor: Highlight the blue  $R$  trace*

6.2. The baseline-corrected  $\Delta R/R$  spectrum showed clear excitonic resonances [1].

6.2.1. LAB MEDIA: Figure 6B. *Video editor: Show the black  $\Delta R/R$  curve*

6.3. The Aspnes third-derivative fit reproduced the  $\Delta R/R$  spectrum [1].

6.3.1. LAB MEDIA: Figure 7A. *Video editor: Keep flashing the cyan curve above the black curve.*

6.4. The model was decomposed into 4 components [1], and their sum was shown by a dashed line [2].

6.4.1. LAB MEDIA: Figure 7B. *Video editor: Highlight the 4 colored curves labelled Fit Peak 1, Fit Peak 2, Fit Peak 3, and Fit Peak 4*

6.4.2. LAB MEDIA: Figure 7B. *Video editor: Highlight the dashed black line labelled cumulative fit peak*

6.5. The  $\Delta R$  line shape was preserved [1] while the amplitude increased monotonically as the alternating-current voltage was increased from 100 to 1200 volts [2].

6.5.1. LAB MEDIA: Figure 8A.

6.5.2. LAB MEDIA: Figure 8A. *Video editor: Sequentially highlight the curves from 100 V curve to the 1200 V curve as the signal becomes larger (dark blue to light blue curves sequentially)*

6.6. The peak magnitudes at selected energies scaled linearly with the applied voltage amplitude [1].

6.6.1. LAB MEDIA: Figure 8B.

6.7. Increasing the lock-in time constant from 3 seconds [1] to 10 seconds [2] and 30 seconds

reduced high-frequency noise and revealed weaker features in molybdenum disulfide [3].

6.7.1. LAB MEDIA: Figure 9A. *Video editor: Highlight the blue curve labelled 3 seconds*

6.7.2. LAB MEDIA: Figure 9A. *Video editor: Highlight the red curve labelled 10 seconds*

6.7.3. LAB MEDIA: Figure 9A. *Video editor: Highlight the gray curve labelled 30 seconds*

### **Pronunciation Guides:**

1. Exfoliated  
Pronunciation link: <https://www.merriam-webster.com/dictionary/exfoliated>  
IPA: /ɪk'sfoʊli,etɪd/  
Phonetic Spelling: ik-sfoh-lee-ay-tuhd
2. Polydimethylsiloxane  
Pronunciation link: <https://www.howtopronounce.com/polydimethylsiloxane>  
IPA: /ˌpɒli,dai,mɛθəl'sɪlɒk,sɪn/  
Phonetic Spelling: pol-ee-dye-meth-uhl-sil-ok-sayn
3. Piezoceramic  
Pronunciation link: <https://www.howtopronounce.com/piezoceramic>  
IPA: /ˌpiːzəʊsə'ræmɪk/  
Phonetic Spelling: pee-zoh-suh-ram-ik
4. Acetone  
Pronunciation link: <https://www.merriam-webster.com/dictionary/acetone>  
IPA: /'æsə,tʊn/  
Phonetic Spelling: as-uh-tohn
5. Adhesive  
Pronunciation link: <https://www.merriam-webster.com/dictionary/adhesive>  
IPA: /əd'hiːsɪv/, /əd'hiːsɪv/  
Phonetic Spelling: ad-hee-siv
6. Monochromator  
Pronunciation link: <https://www.howtopronounce.com/monochromator>  
IPA: /ˌmɒnəʊ'krəʊmə,tetər/  
Phonetic Spelling: mon-oh-kroh-muh-tay-ter
7. Photodiode  
Pronunciation link: <https://www.merriam-webster.com/dictionary/photodiode>  
IPA: /'fəʊtəʊ,daiəʊd/  
Phonetic Spelling: foh-toh-dye-ohd
8. Preamplifier  
Pronunciation link: <https://www.merriam-webster.com/dictionary/preamplifier>  
IPA: /pri:'æmplə,faiər/  
Phonetic Spelling: pree-am-pluh-fy-er

9. Oscilloscope  
Pronunciation link: <https://www.merriam-webster.com/dictionary/oscilloscope>  
IPA: /əˈsɪləˌskoʊp/  
Phonetic Spelling: uh·sil·uh·skohp
10. Monochromatic  
Pronunciation link: <https://www.merriam-webster.com/dictionary/monochromatic>  
IPA: /ˌmɒːnɒkrəʊˈmætɪk/  
Phonetic Spelling: mon·oh·kroh·mat·ik
11. Halogen  
Pronunciation link: <https://www.merriam-webster.com/dictionary/halogen>  
IPA: /ˈhælədʒən/  
Phonetic Spelling: hal·uh·juhn
12. Chopper  
Pronunciation link: <https://www.merriam-webster.com/dictionary/chopper>  
IPA: /ˈtʃɒːpər/  
Phonetic Spelling: chop·er
13. Reflectance  
Pronunciation link: <https://www.merriam-webster.com/dictionary/reflectance>  
IPA: /rɪˈflektəns/  
Phonetic Spelling: ri·flek·tuhns
14. Aspnes  
Pronunciation link: <https://www.howtopronounce.com/aspnes>  
IPA: /ˈæspnɛz/  
Phonetic Spelling: asp·nez
15. Resonance  
Pronunciation link: <https://www.merriam-webster.com/dictionary/resonance>  
IPA: /ˈrezənəns/  
Phonetic Spelling: rez·uh·nuhns