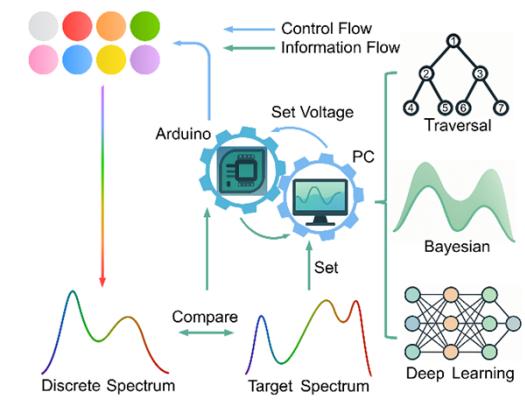
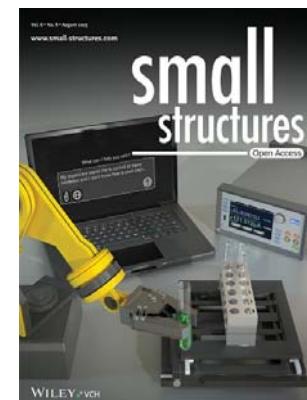
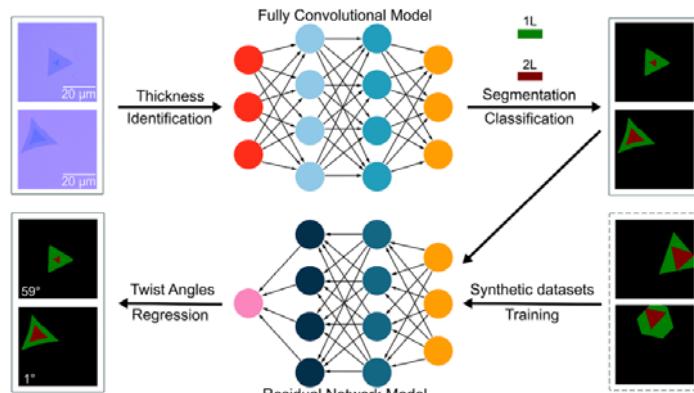


Towards Full Autonomous Synthesis and Characterization of 2D Materials

Yong Xie

Instituto de Ciencia de Materiales de Madrid
(ICMM-CSIC), Spain
xieyong.nwpu@gmail.com



- Motivation
- Deep learning for recognition of twisted angles of CVD-grown 2D materials
- Toward fully autonomous laboratory instrumentation control with large language model
- Toward fully autonomous laboratory
- Take home message

The concept of autonomous (self-driving) lab

Can we make everything autonomously?



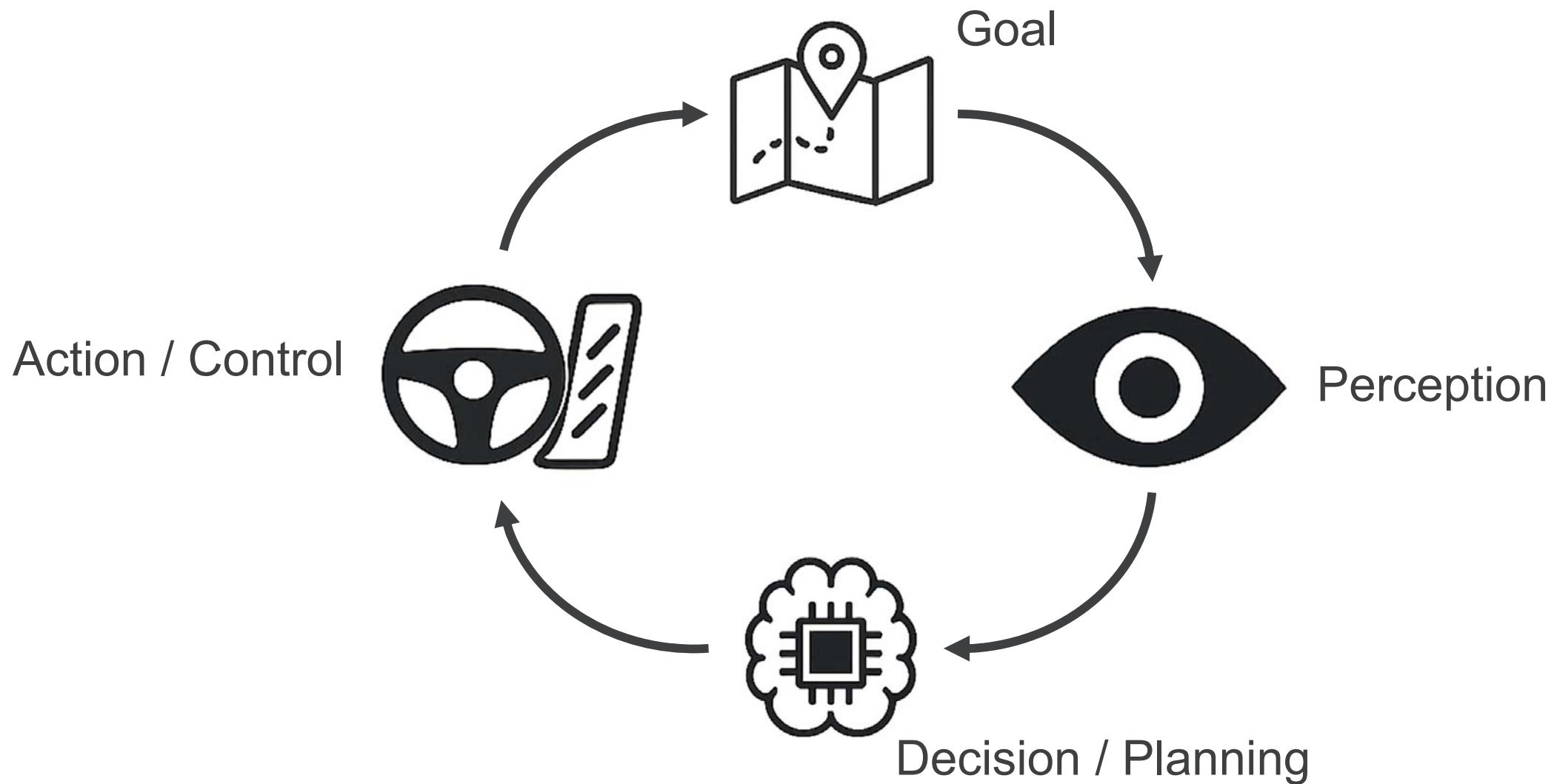
Create/Have a **Doraemon**

Doraemon is a famous Japanese cartoon about a blue robotic cat from the future who helps a boy fulfill **all his wishes**.

Doraemon (ドラえもん) is a Japanese [manga](#) series written and illustrated by [Fujiko F. Fujio](#)

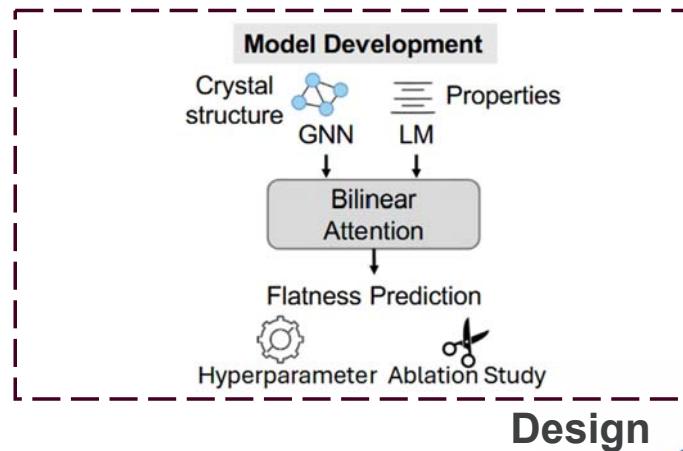


The concept of autonomous (self-driving) lab



The concept of autonomous (self-driving) lab for 2D Mat.

A. Mishchenko et. al,
Structure-Informed
Learning of Flat Band
2D Materials
arXiv:2506.07518

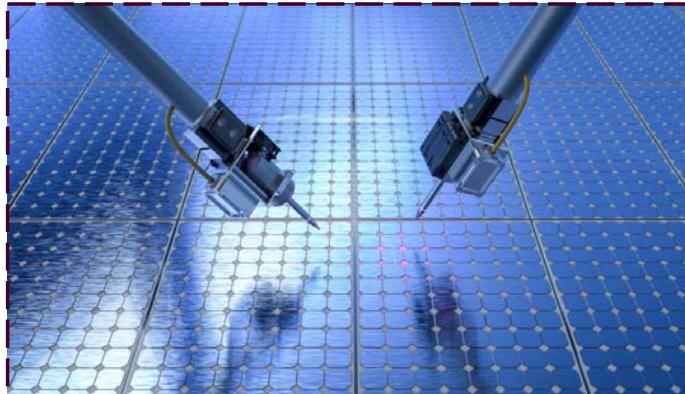


Graph neural networks and language models guide **inverse design** of 2D materials, predicting flat-band candidates from crystal structures.

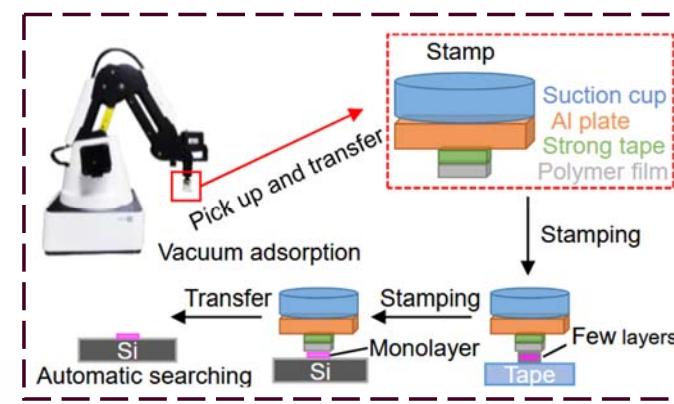
Design

Robotic assembly and **automated** probing accelerate device prototyping, ensuring precise stacking and scalable integration.

Devices



T. Buonassisi et. al, A self-supervised robotic system for autonomous contact-based spatial mapping of semiconductor properties
Sci. Adv. 11, eadw7071 (2025)

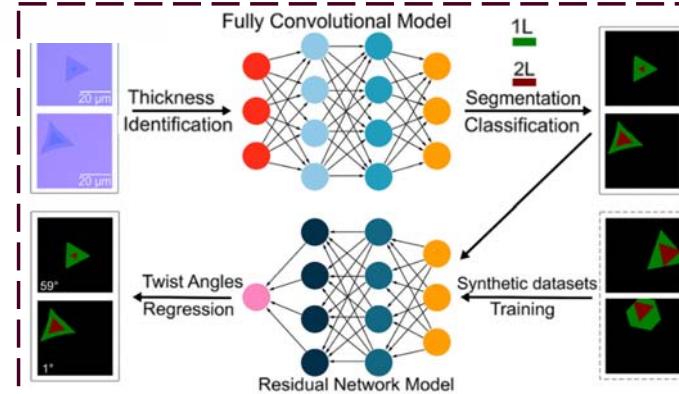


Materials Synthesis

Autonomous robotic exfoliation with **Bayesian optimization (BO)** enables reproducible fabrication of monolayer and few-layer 2D crystals.

AI-driven image analysis **rapidly identifies** layer thickness, stacking, and twist angles from microscopy data.

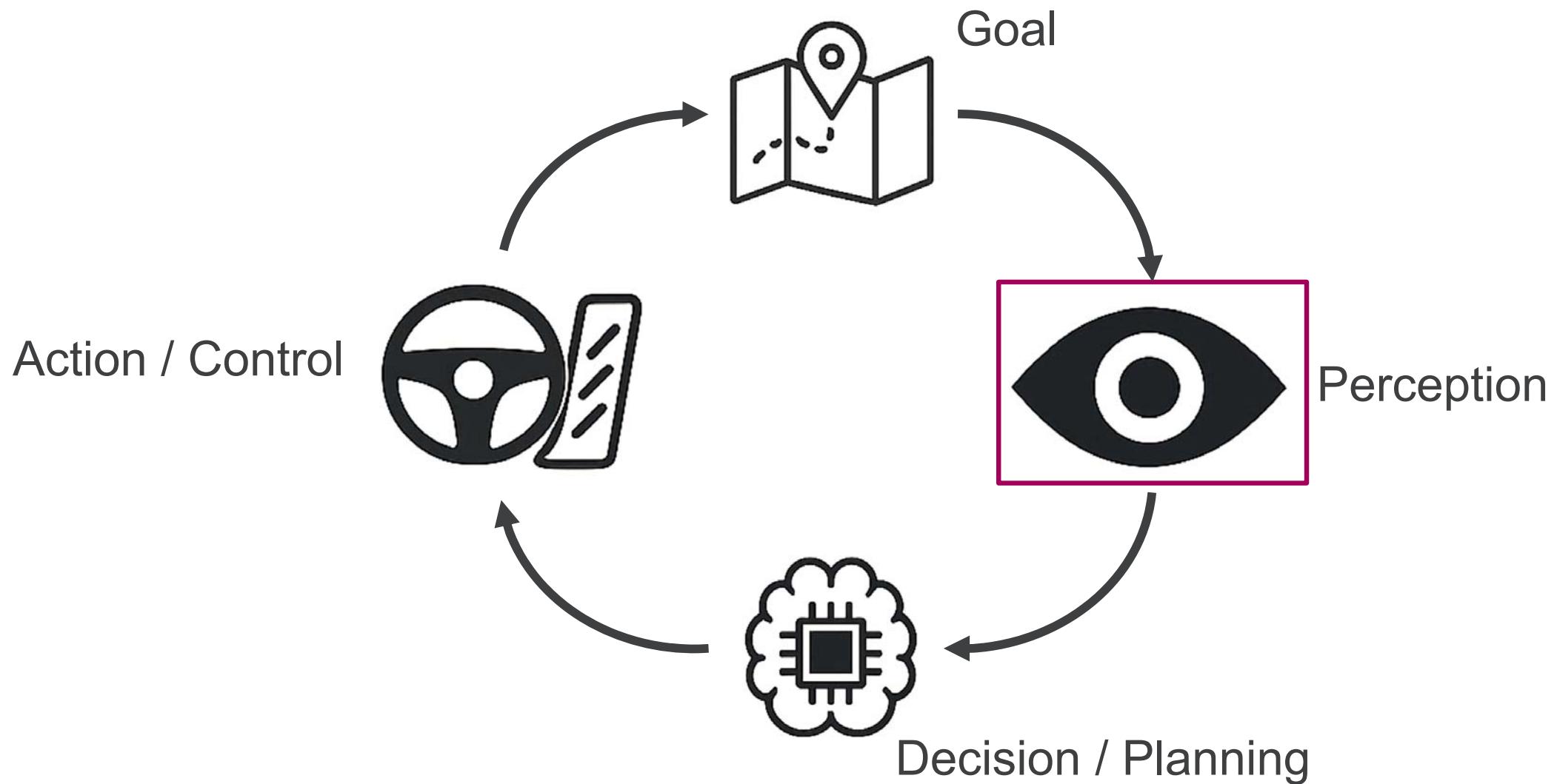
Materials Characterization



K. Matsuda et. al,
Autonomous robotic mechanical exfoliation of two-dimensional semiconductors combined with Bayesian optimization
arXiv: 2411.06891v1

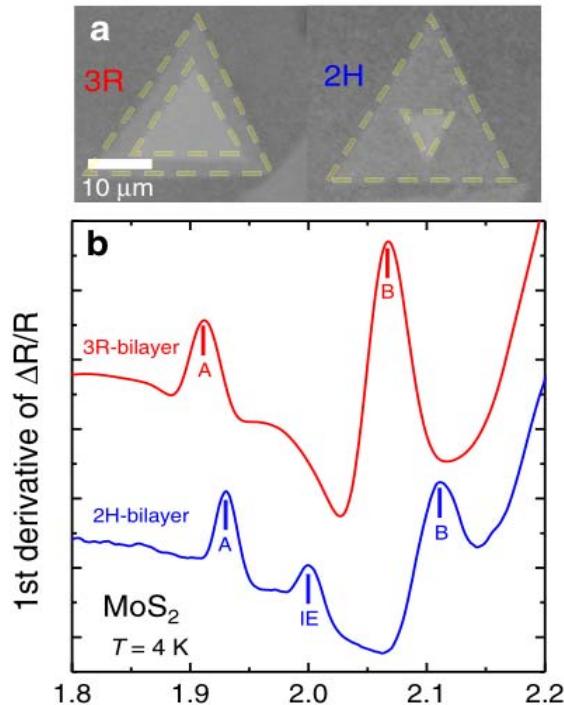
Y. Xie et. al, Identification and structural characterization of twisted atomically thin bilayer materials by deep learning
Nano Lett. 24 (9), 2789-2797 (2024)

The concept of autonomous (self-driving) lab

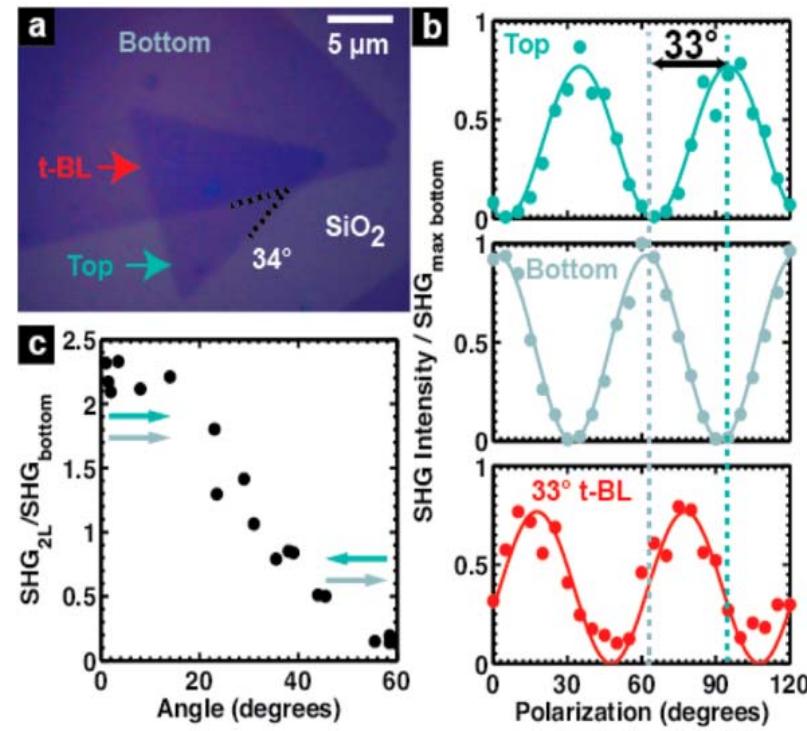


Motivation

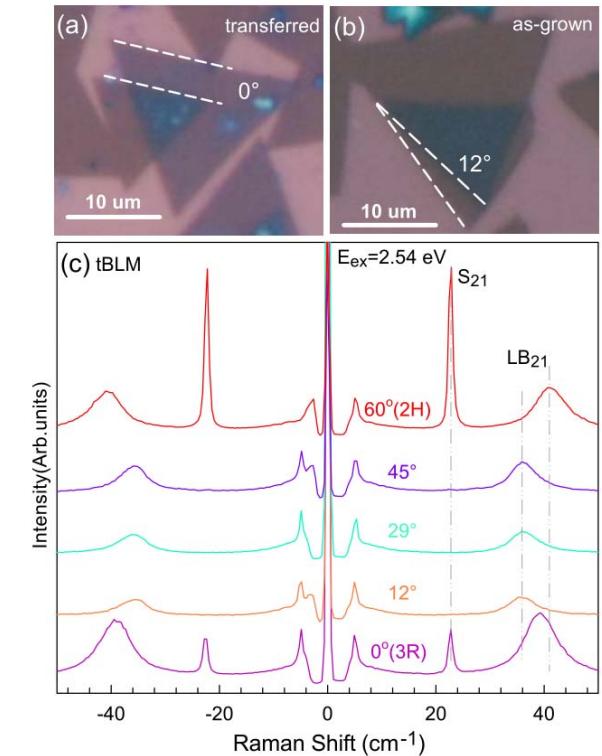
The current measurement methods for twist angles in bilayer TMDs



Differential Reflectance [1]



Second-harmonic generation [2]



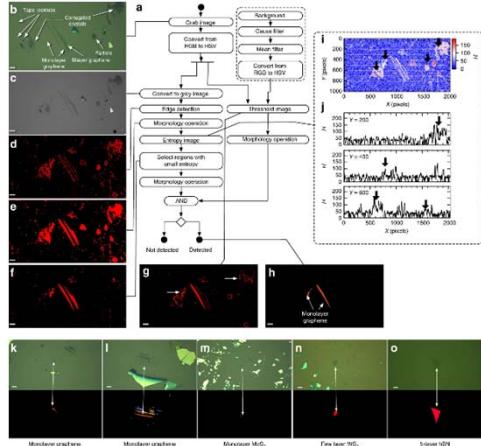
Low wavenumber Raman spectra [3]

[1] *Nat Commun* 11, 2391 (2020)

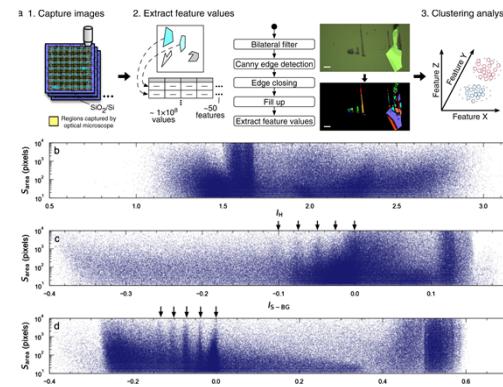
[2] *Nano Lett.* 2014 Jul 9;14(7):3869-75.

[3] *ACS Nano* 2018, 12, 8, 8770–8780

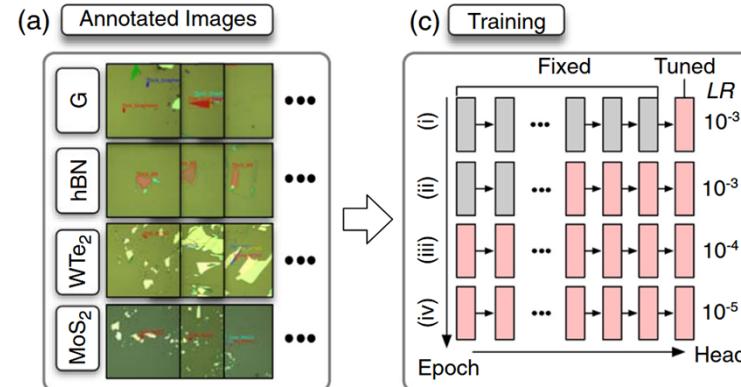
Motivation



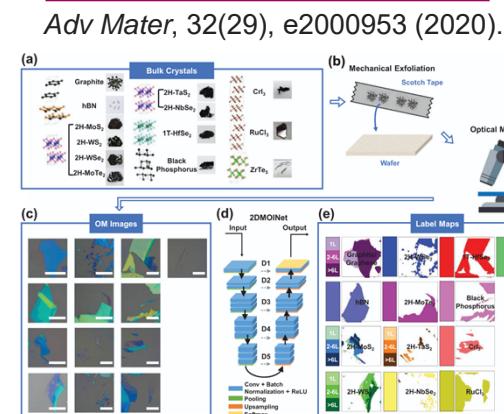
Nat Commun 9, 1413 (2018)



npj 2D Mater Appl 3, 4 (2019).

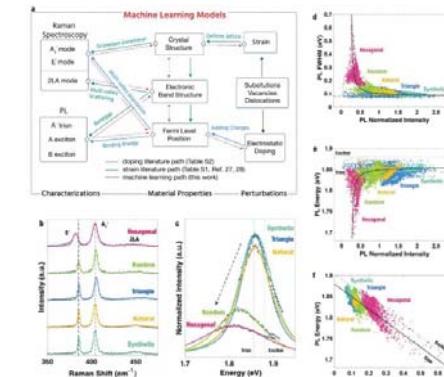


npj 2D Mater Appl 4, 3 (2020).



Adv Mater, 32(29), e2000953 (2020).

Adv Mater, 34(34), 2202911 (2022)

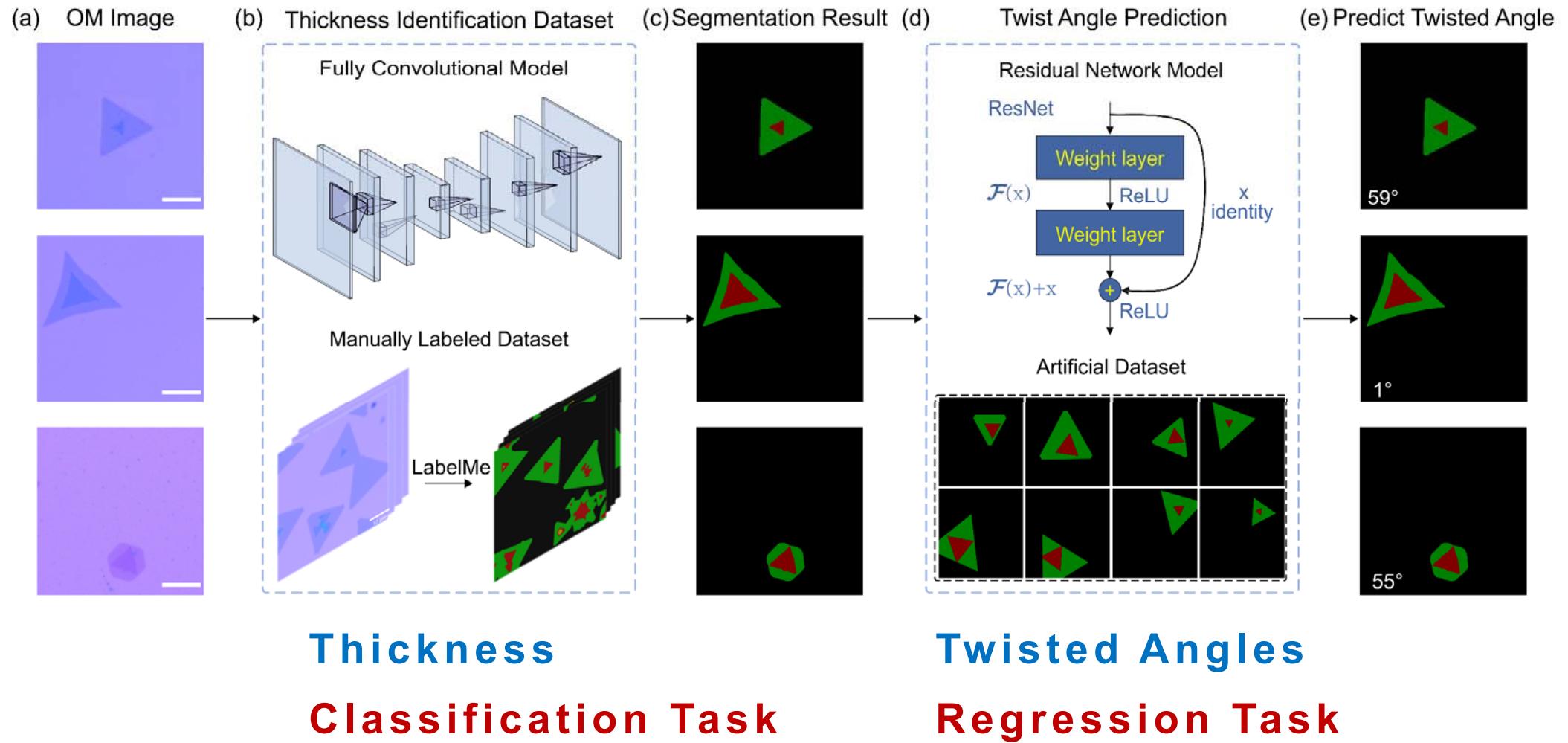


The identification of **contrast**, **color**, **edges**, **shapes**, **flake sizes**, and their **distributions** is crucial for advancing research on 2D materials and their device applications.

What's the main difficulty in the design of the AI (NN) to recognize the twisted angle of 2D materials?

Enough Datasets?

Two-step process



Data Preparation (CVD growth process)



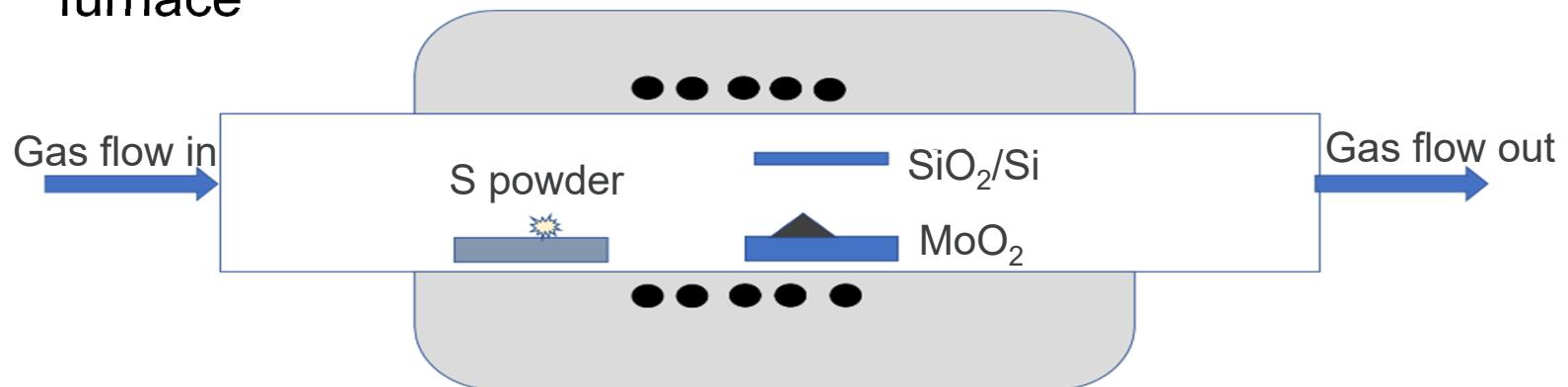
Thermo Scientific
Single-zone tube
furnace



Alicat Scientific
Gas flow control valve

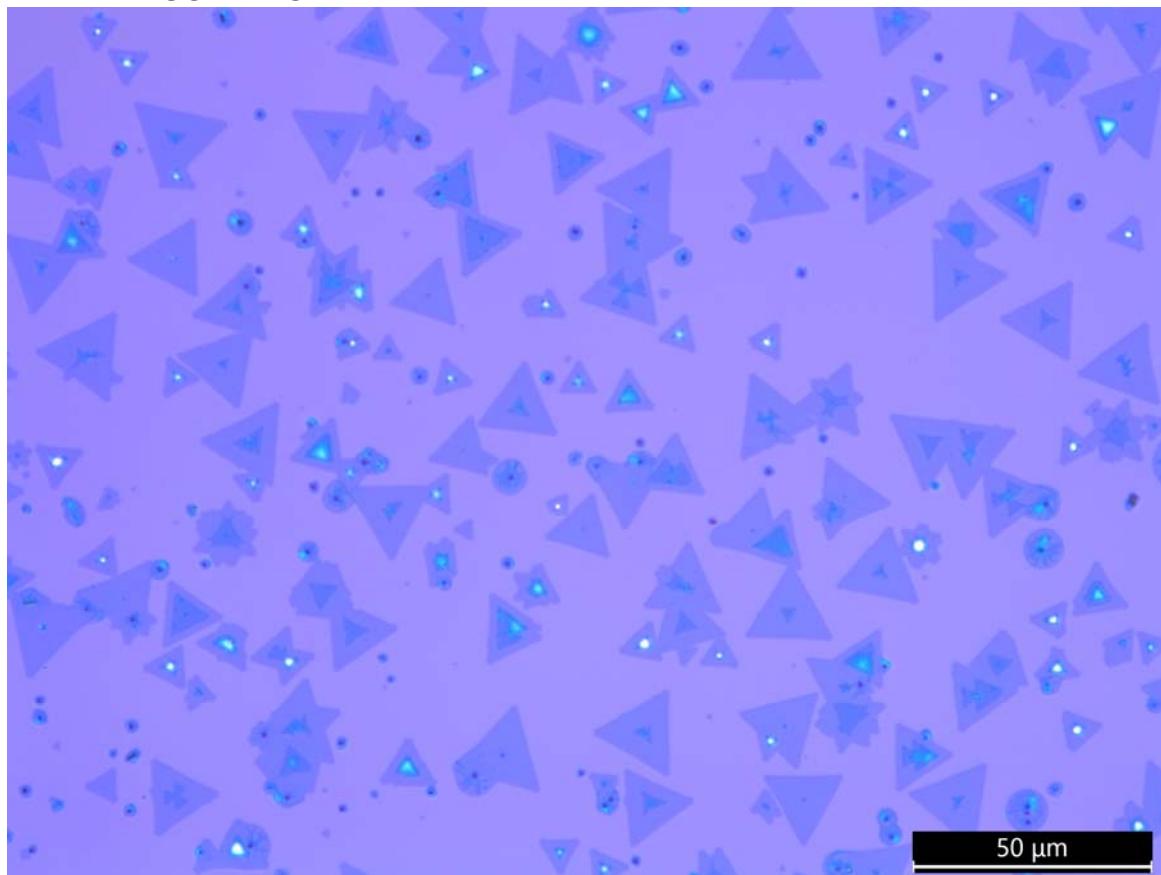


Ar Supply



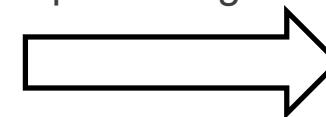
Data preprocessing

2592×1944



In total 1035 micrograph images

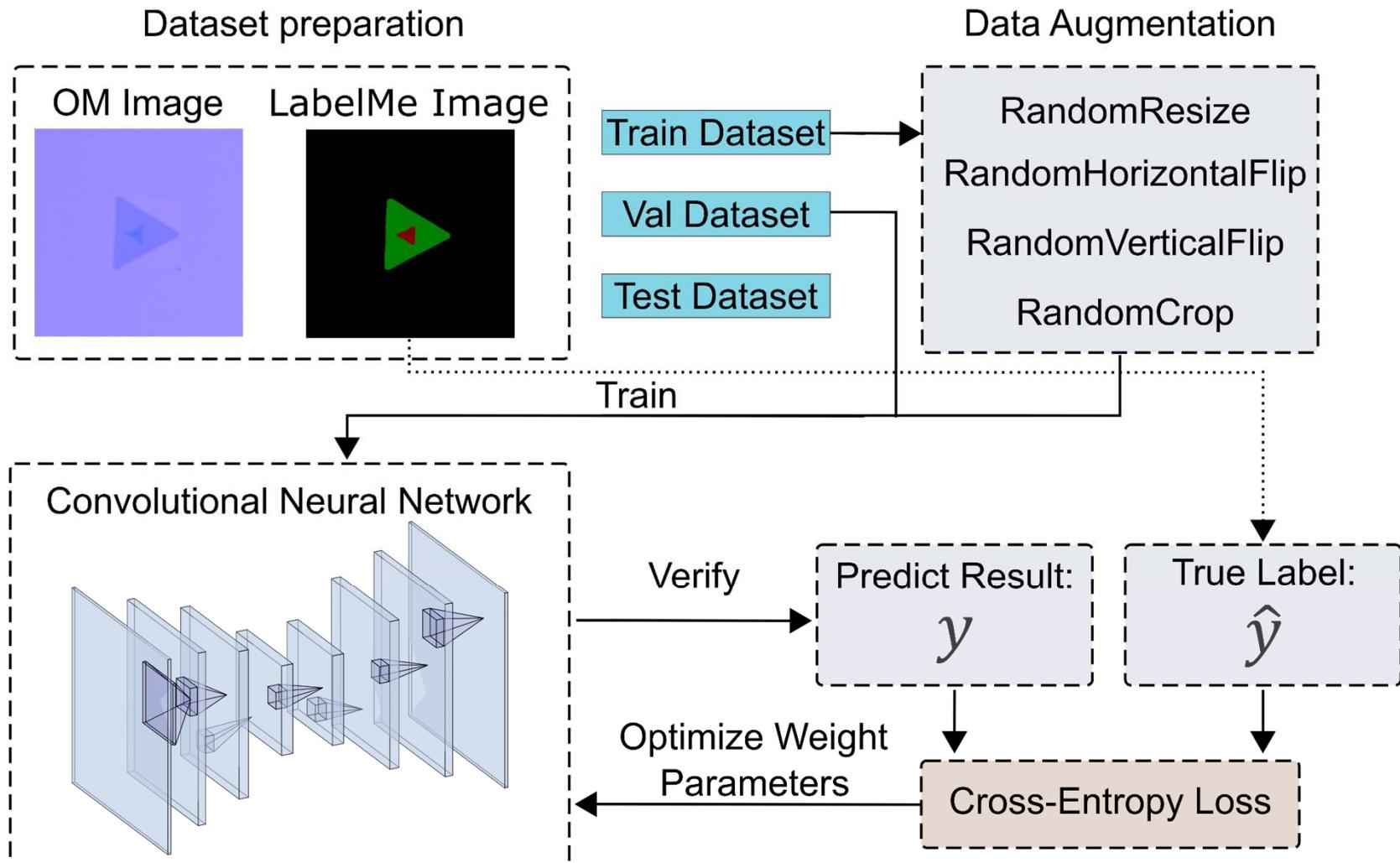
512×512
pixel images



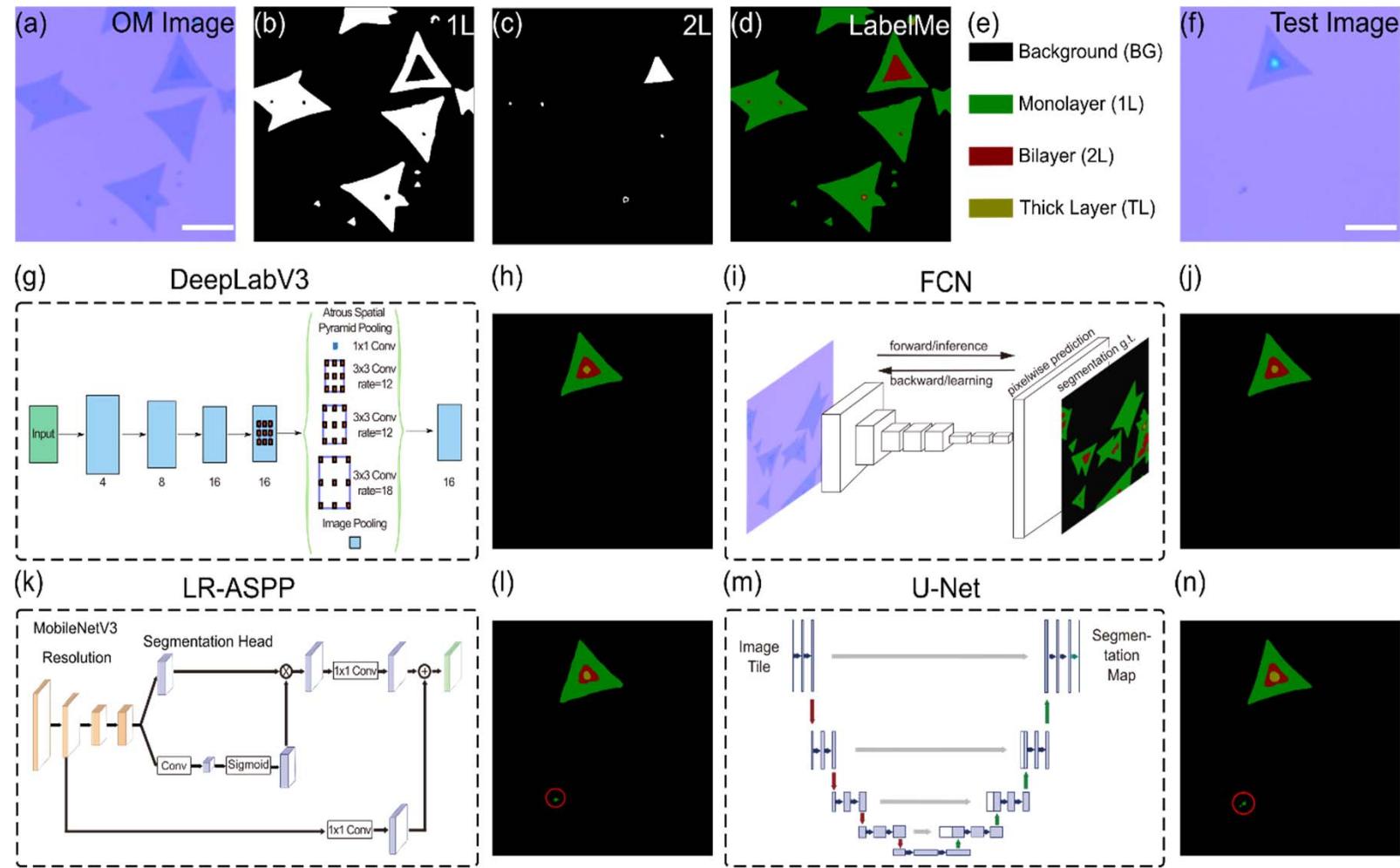
Datasets



Deep Learning to Identify the Thickness of TMDs

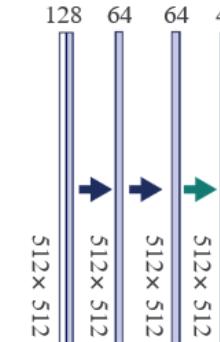
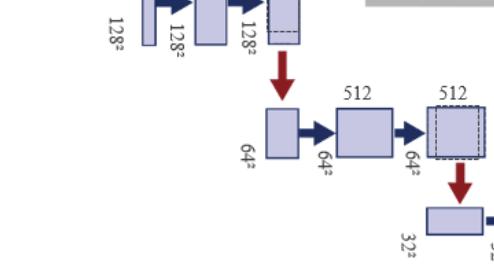
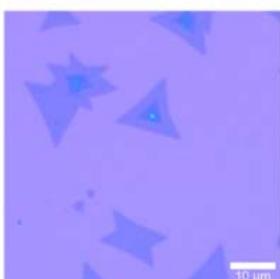
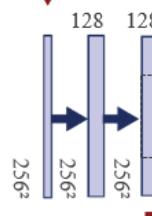
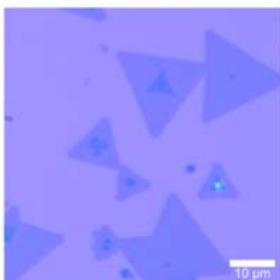
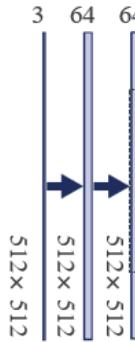
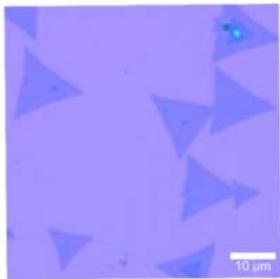


Deep Learning to Identify the Thickness of TMDs

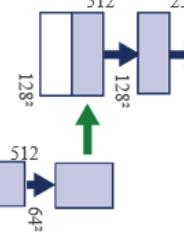
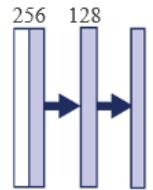
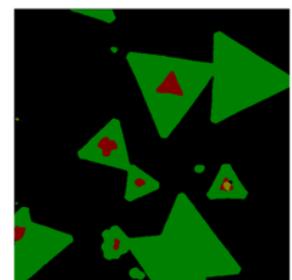
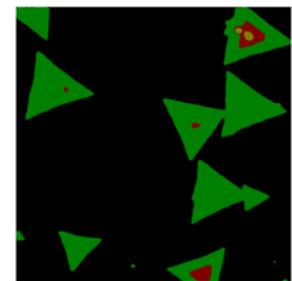


Deep Learning to Identify the Thickness of TMDs

Input Images



Output Images

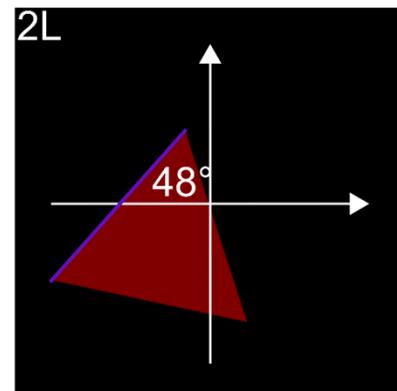
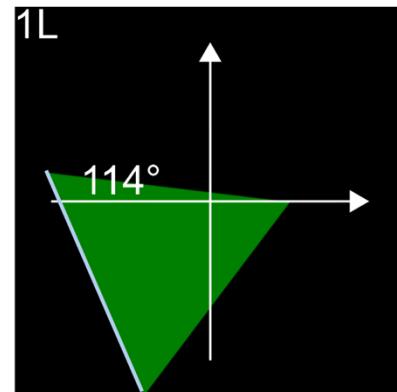


Deep Learning to identify Twist Angles

1.Find the leftmost side of each triangle.

2.Determine its rotation angle relative to the center of the image

3.Calculate the torsion angle based on the rotation angle of the leftmost side of the single and double layers.

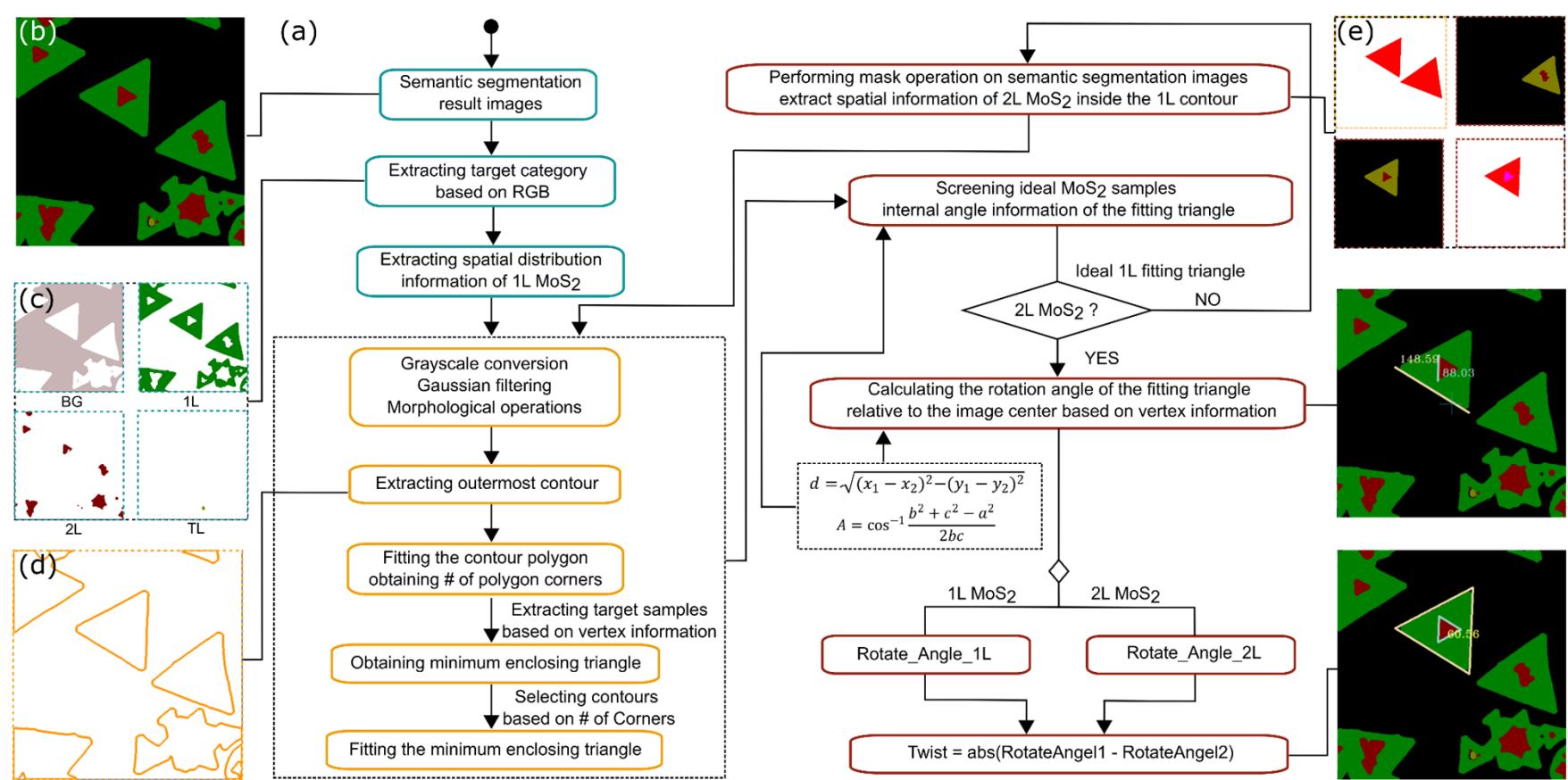


OpenCV to Identify Twisted Angles of TMDs

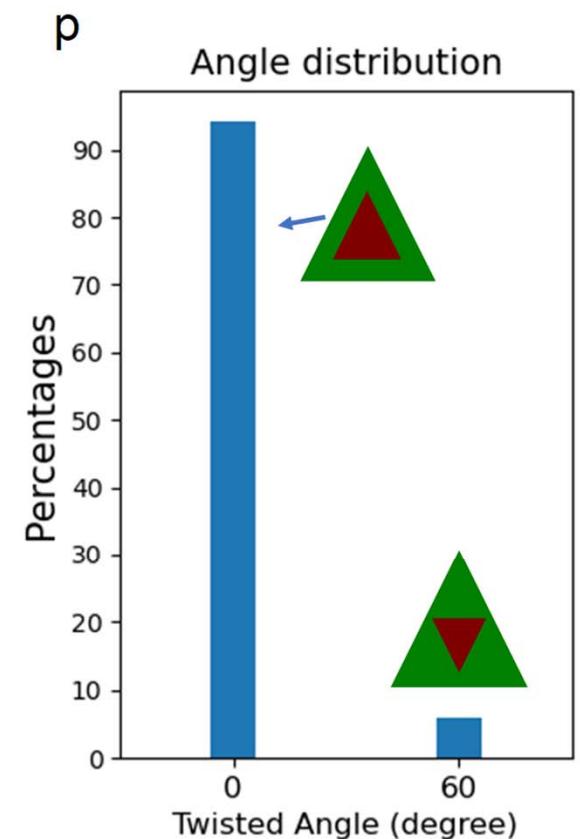
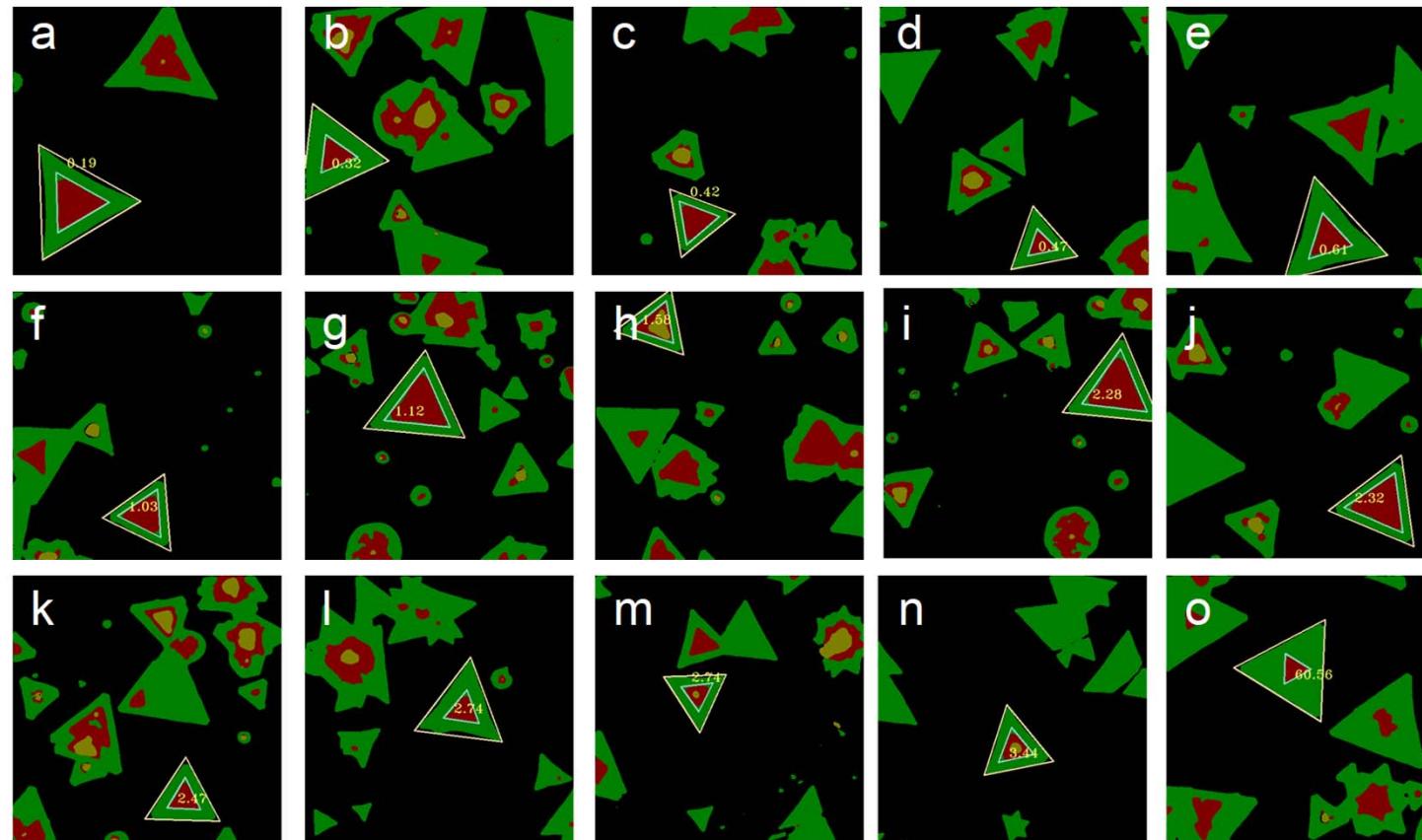
Simplified workflow diagram



Detailed workflow diagram



OpenCV to Predict the Twisted Angles of TMDs



Half intelligence

MIT and Toyota release innovative dataset to accelerate autonomous driving research

DriveSeg contains precise, pixel-level representations of many common road objects, but through the lens of a continuous video driving scene.

MIT AgeLab
June 18, 2020

Tesla CEO Elon Musk says at least 9.6 billion kilometers are needed, while the RAND Corporation believes at least 17.7 billion kilometers are required — equivalent to more than **50 round trips between Earth and the Sun**. With such a massive amount of data, if it's all collected through road testing, wouldn't that drive engineers crazy?



Use **synthetically generated datasets** to simulate autonomous driving environments for training models.

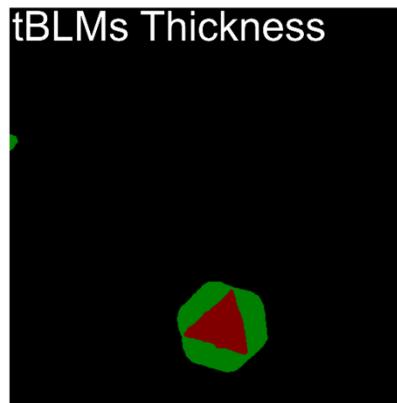
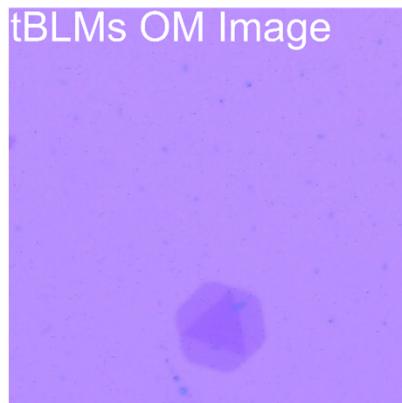
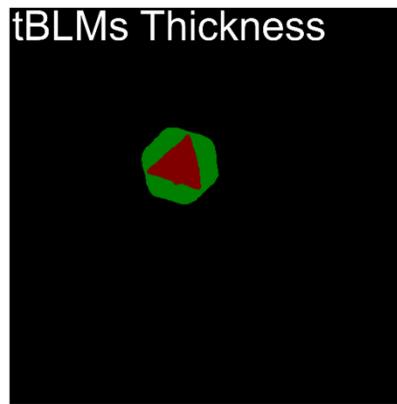
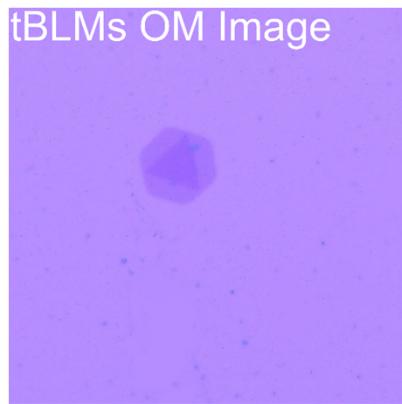


<https://news.mit.edu/2020/mit-toyota-release-visual-open-data-accelerate-autonomous-driving-research-0618>

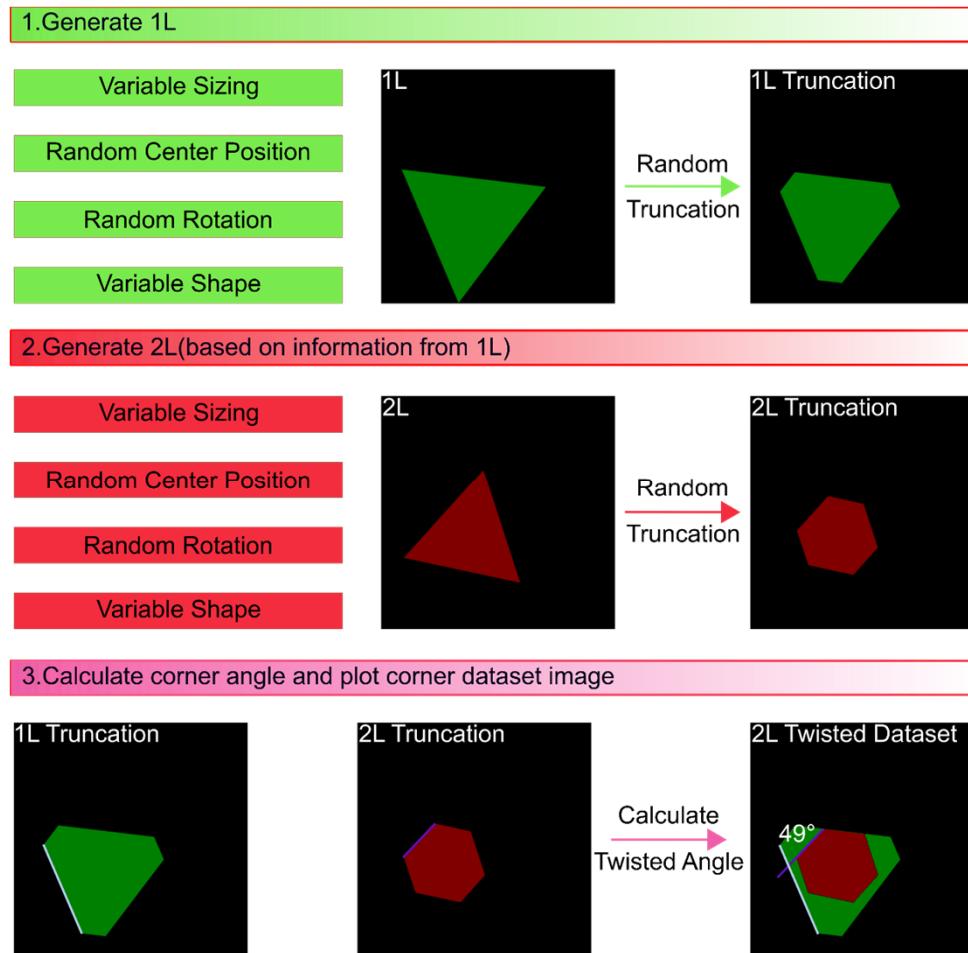
Training Dataset Preparation



True Datasets

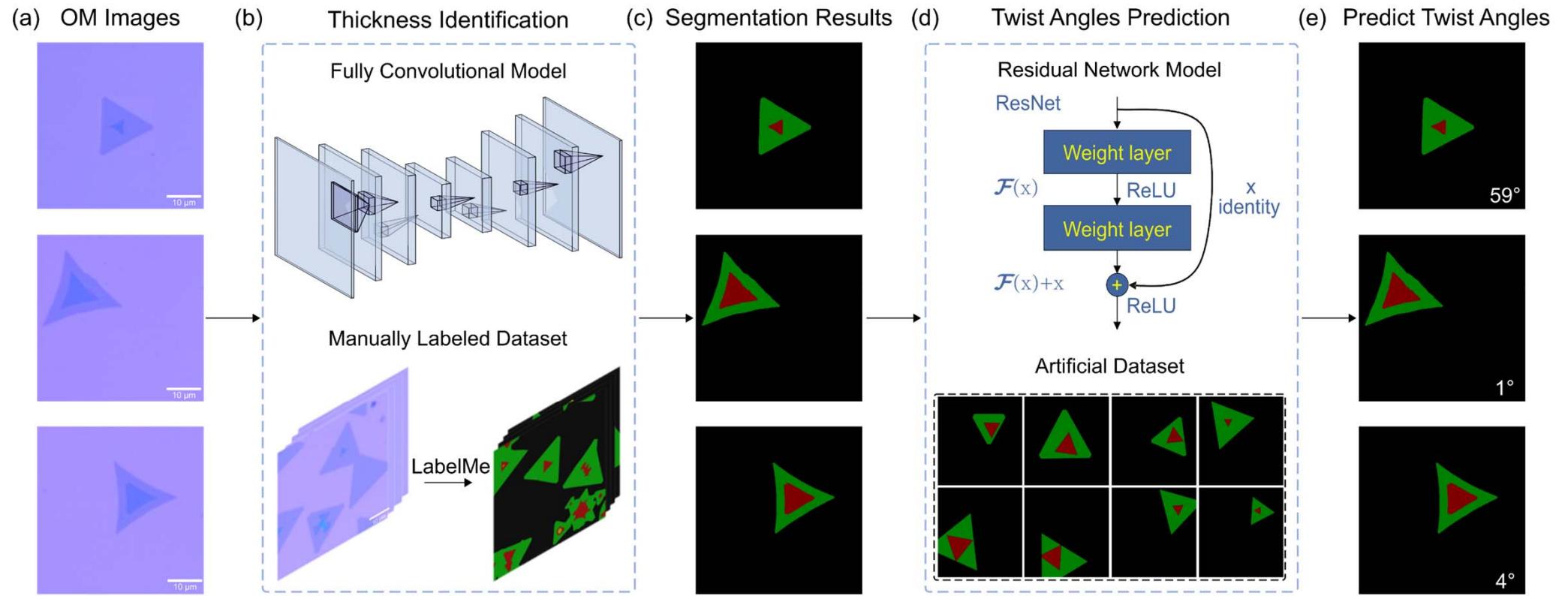


Artificial Datasets



Eduardo
R. Hernandez

Deep Learning to Predict Twist Angles



Full intelligence

(d) Twist Angles Prediction

Residual Network Model

ResNet

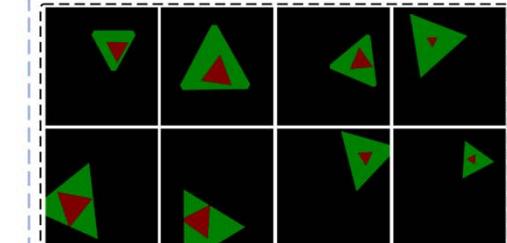
Weight layer

Weight layer

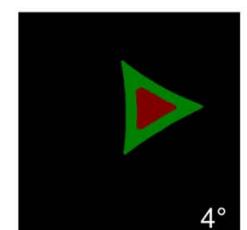
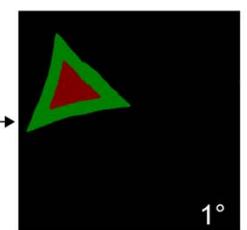
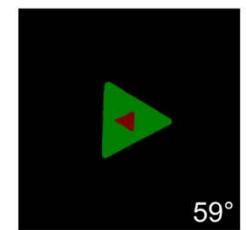
$\mathcal{F}(x)$

$\mathcal{F}(x)+x$

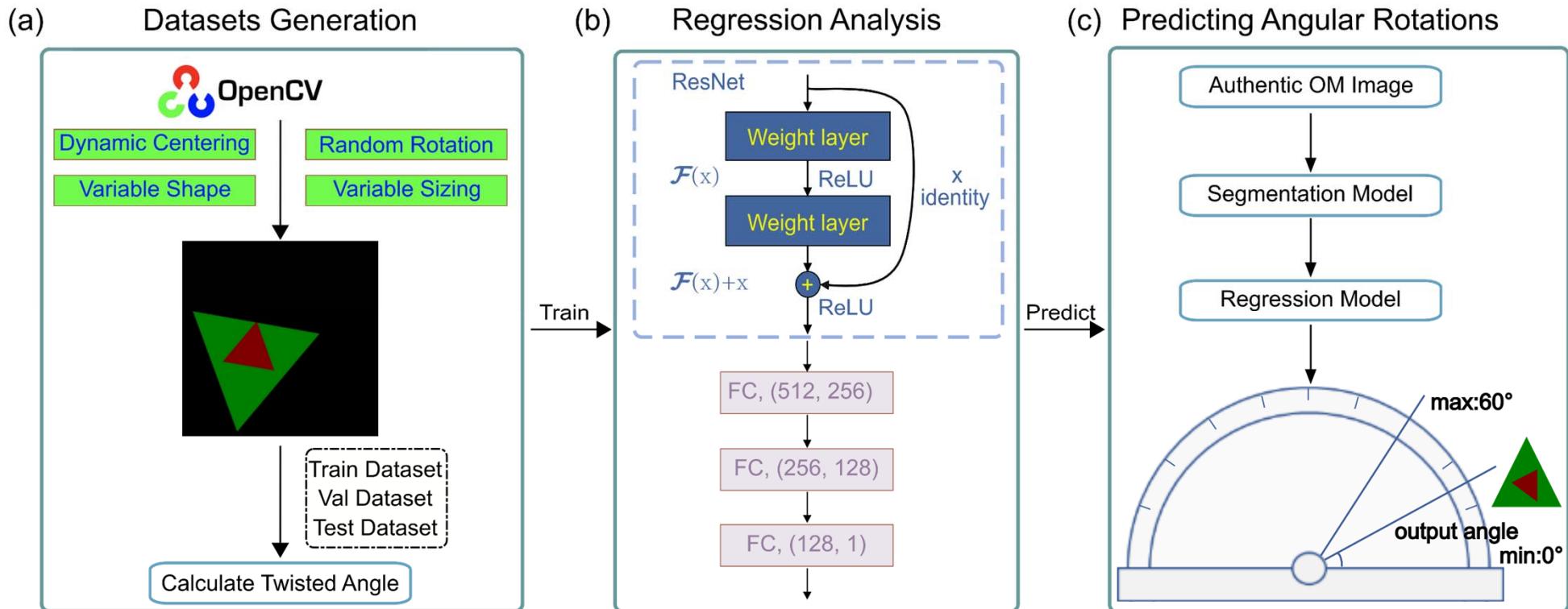
Artificial Dataset



(e) Predict Twist Angles



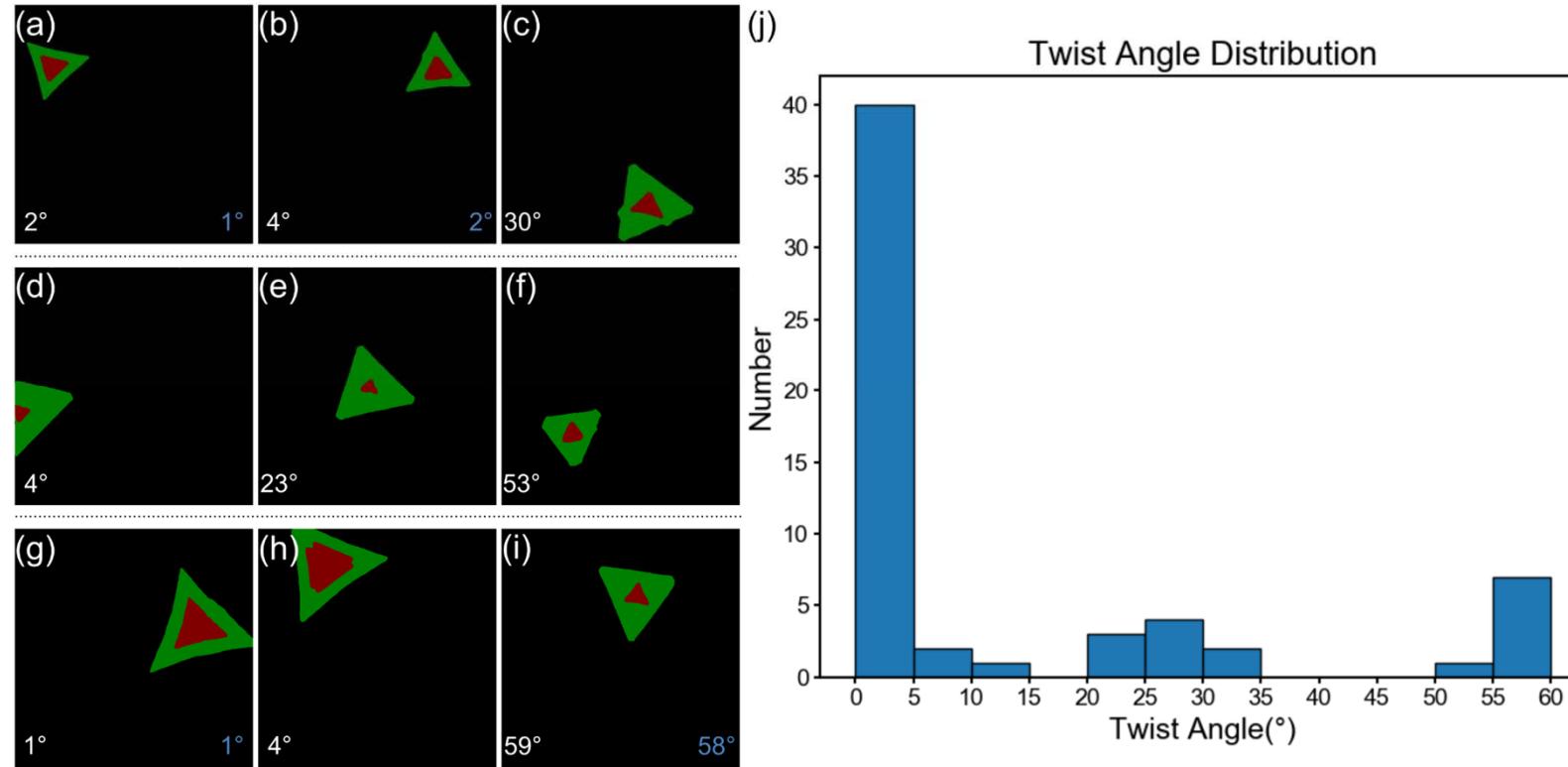
Deep Learning Predict Twist Angles



Deep learning approach for recognizing twist angles in MoS_2 flakes.

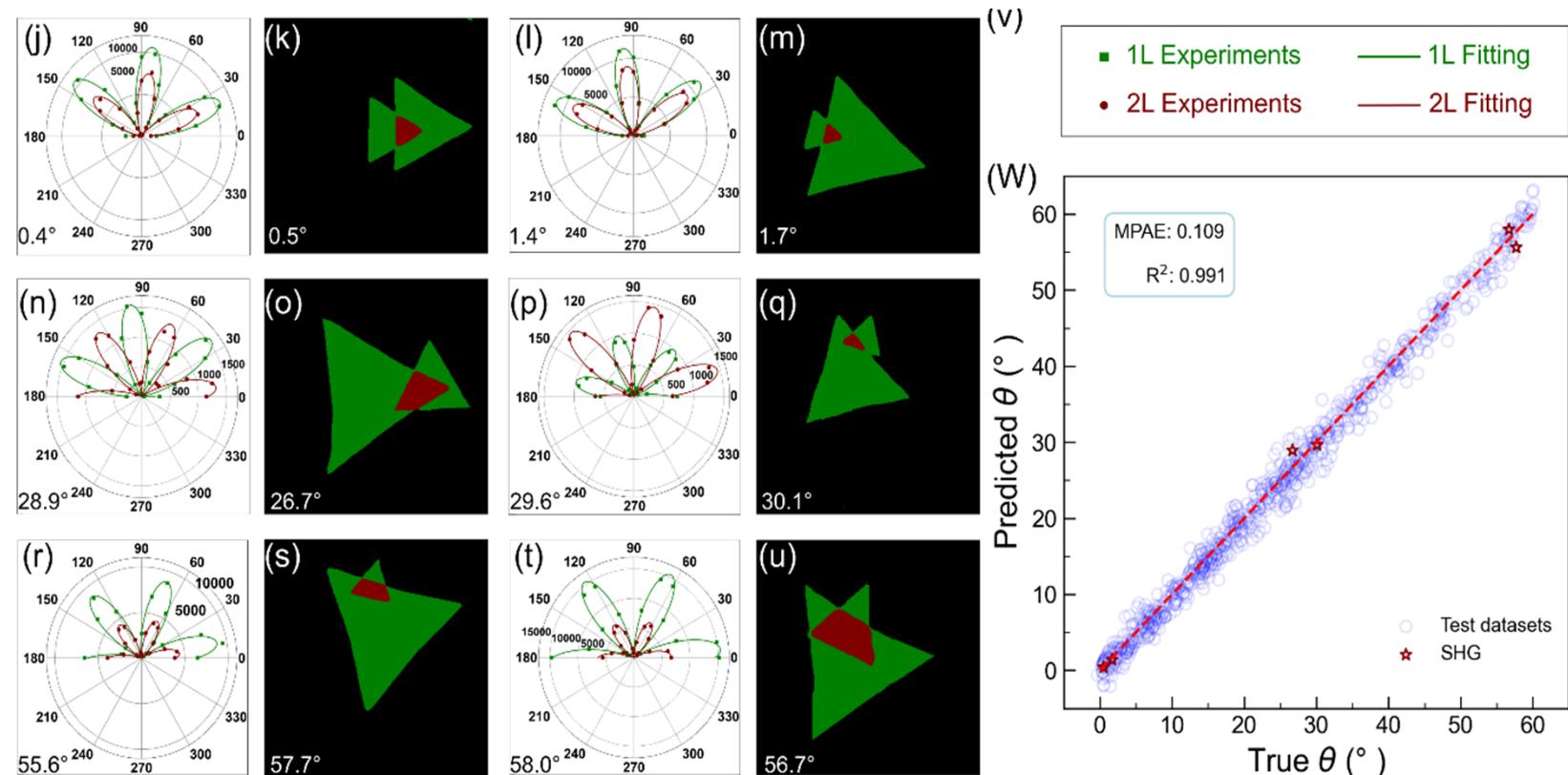
- (a) Synthetic dataset illustrating varying twist angles in uniformly colored MoS_2 flakes post-segmentation.
- (b) ResNet CNN model training using the linear regression approach on the dataset from (a).
- (c) Prediction of twist angles for actual as-grown MoS_2 bilayer samples post-segmentation

Deep Learning Predict Twist Angles



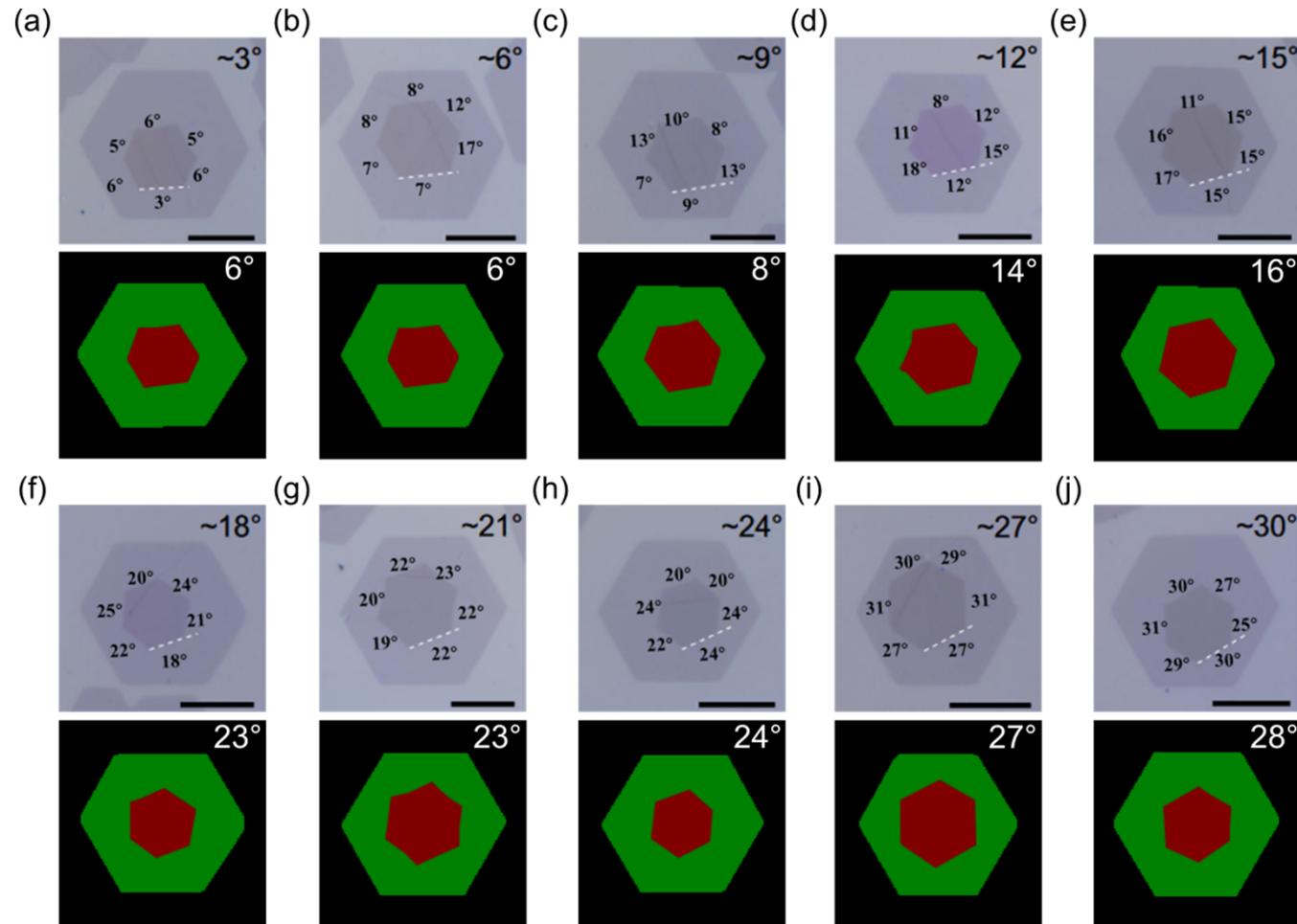
Performance evaluation of the twist angle Identification Model.

Deep Learning to Predict Twist Angles

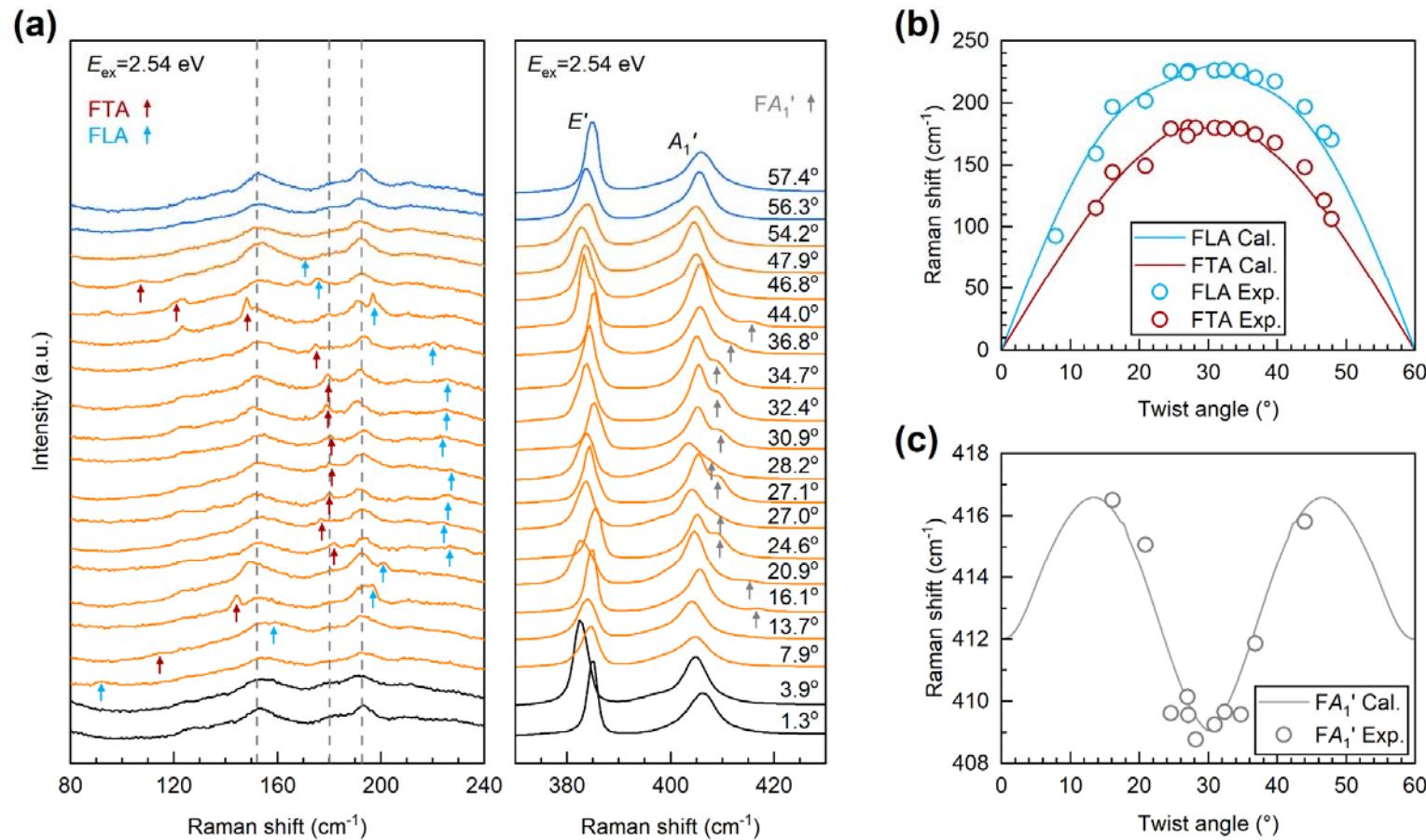


Pingheng
Tan

Deep Learning to Predict Twist Angles of Bilayer Graphene



Moiré phonons in twisted CVD grown bilayer MoS₂

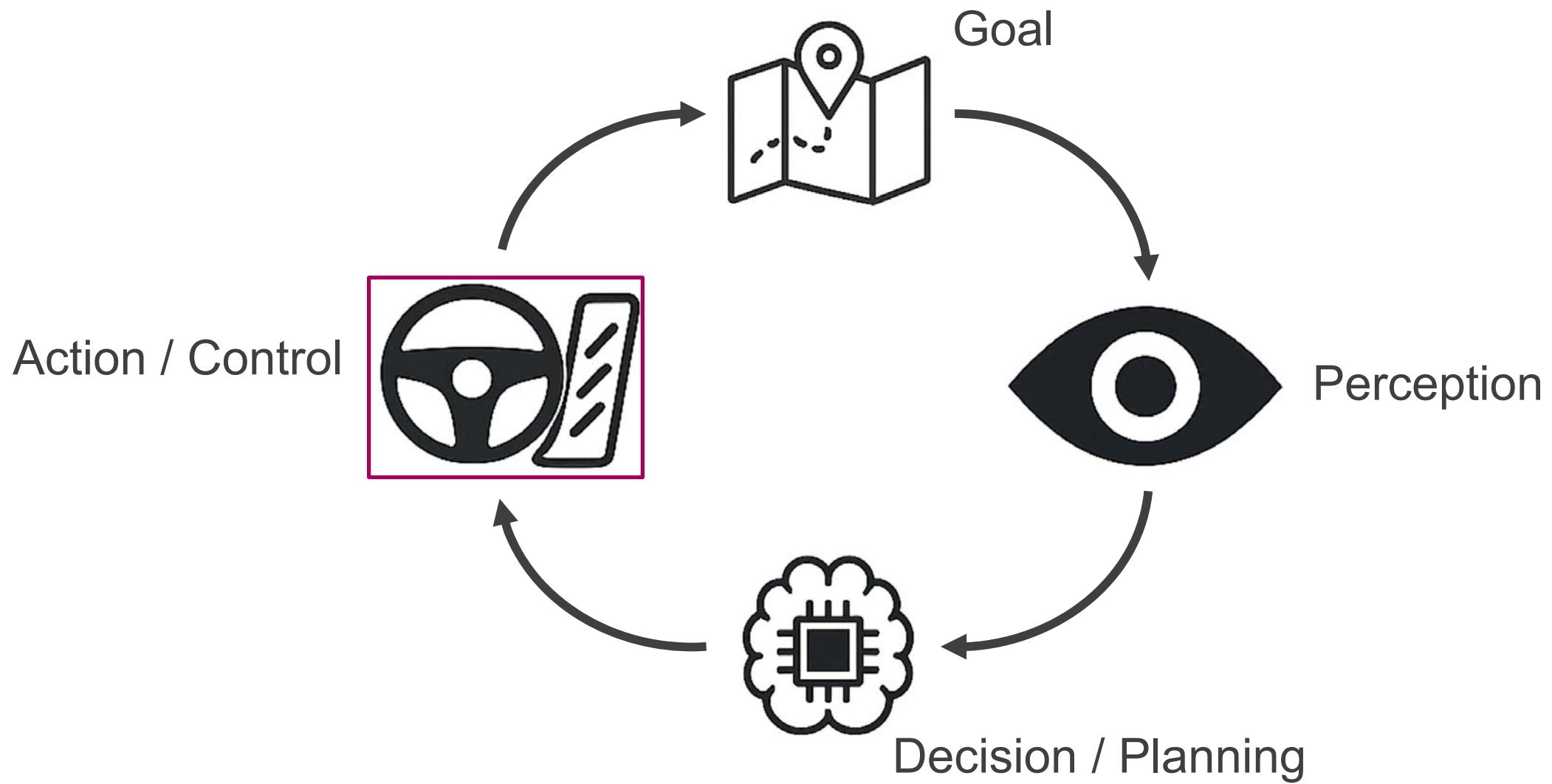


Pingheng
Tan

Deep Learning to Predict Twist Angles

		Thickness Classification Model				Twist Angle Regression Model
Model Name		DeepLabV3	FCN	LR-ASPP	U-Net	ResNet
Training Epoch		300	300	300	300	600
NN Training Time		29m24s	25m18s	17m18s	34m18s	14h25m33s
Frames Per Second (FPS)	CPU	1.16	1.32	9.80	3.56	14.3
	GPU	45.66	50.35	201.25	125.63	479.6

The concept of autonomous (self-driving) lab



Towards Autonomous Laboratory Instrumentation Control with LLM-based Tools



“Standard”

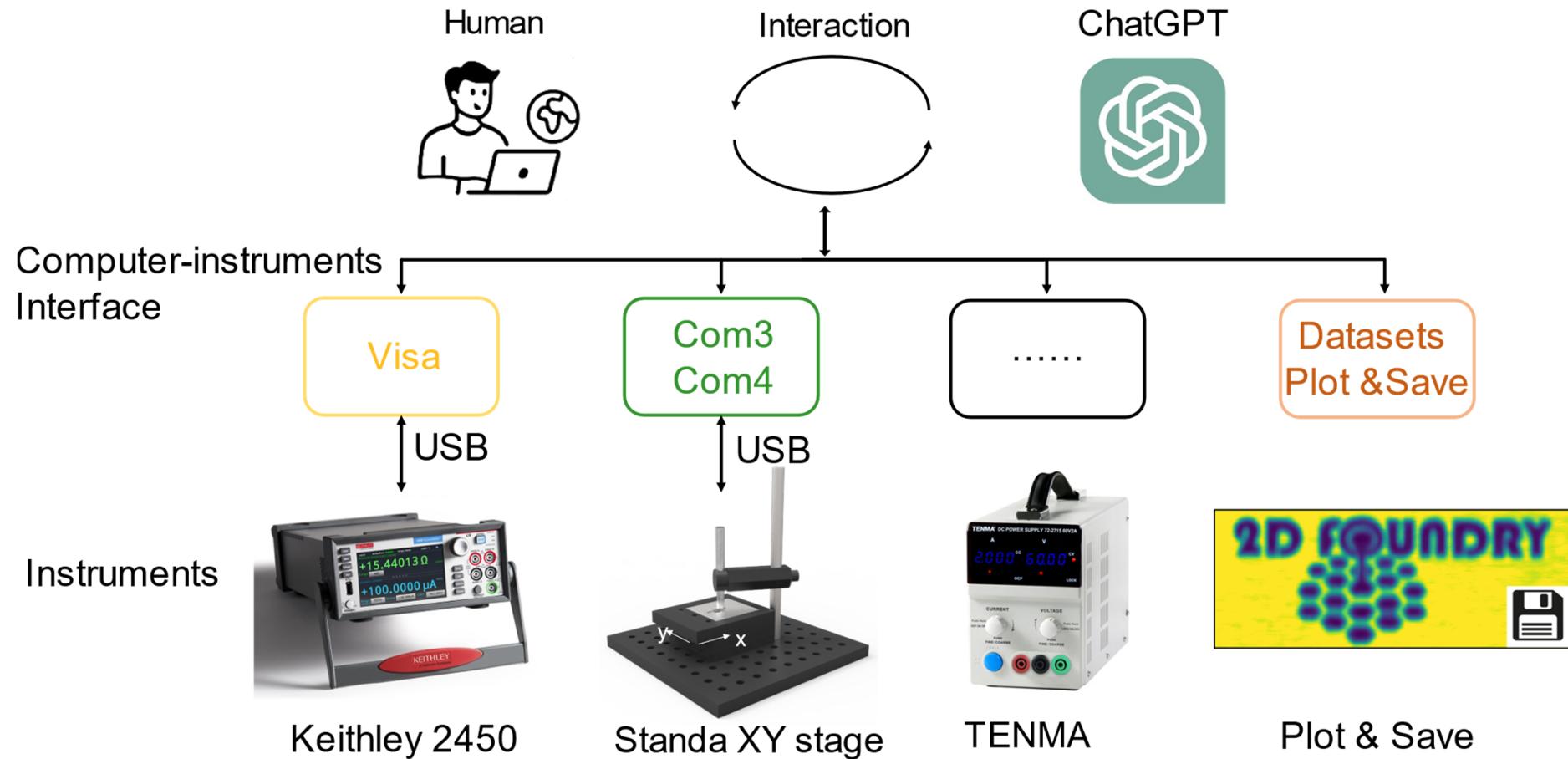


“Non – Standard”



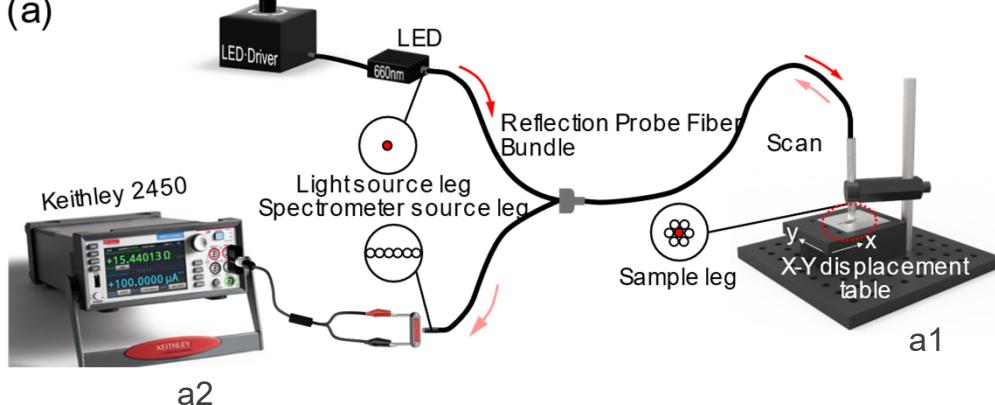
software developer

Towards autonomous laboratory instrumentation control with LLM-based tools



Towards autonomous laboratory instrumentation control with LLM-based tools

(a)



a1

Move with the XY stage

a2

Measure the current with applying voltage with the Keithley

a3

Single pixel camera
Raster/scan mapping

a4

Data Plot & Save

b1

Prompt 1-input

Write MATLAB code to initialize and set up a Standa XY stage with the serial number 8SMC4-USB-B9-2 using the libximc library. The XY stage use the com3 and com4 ports. Can you write from the **very basic, simple code, and step by step, stop at the every step for me to confirm it works (and give me the feedback of it is successfully if it works, then can process to the next step) to reach the goal here for me?** Clear all the connections and make sure give a fresh start before the measurement! Use the following code snippet to load the library (Please do not change this part), ensuring compatibility with a 64-bit Windows system

b2

Prompt 2-input

Write MATLAB code to control a Keithley 2450 SourceMeter using the VISA interface. The code should connect to the instrument using its VISA resource name, reset it, configure it to source a specific voltage (e.g., 0.01 V), and set it to measure DC current... Can you write from the **very basic, simple code, and step by step, stop at the every step for me to confirm it works (and give me the feedback of it is successfully if it works) to reach the goal here for me?** Clear all the connections and make sure give a fresh start before the measurement!

b3

Prompt 3-input

Control of the Standa to move and measure the current at each spot with the same voltage of 0.1V. Then plot the results afterwards. Can you write from the **very basic, simple code, and step by step, stop at the every step for me to confirm it works (and give me the feedback of it is successfully if it works, then can process to the next step) to reach the goal here for me?** Clear all the connections and make sure give a fresh start before the measurement!

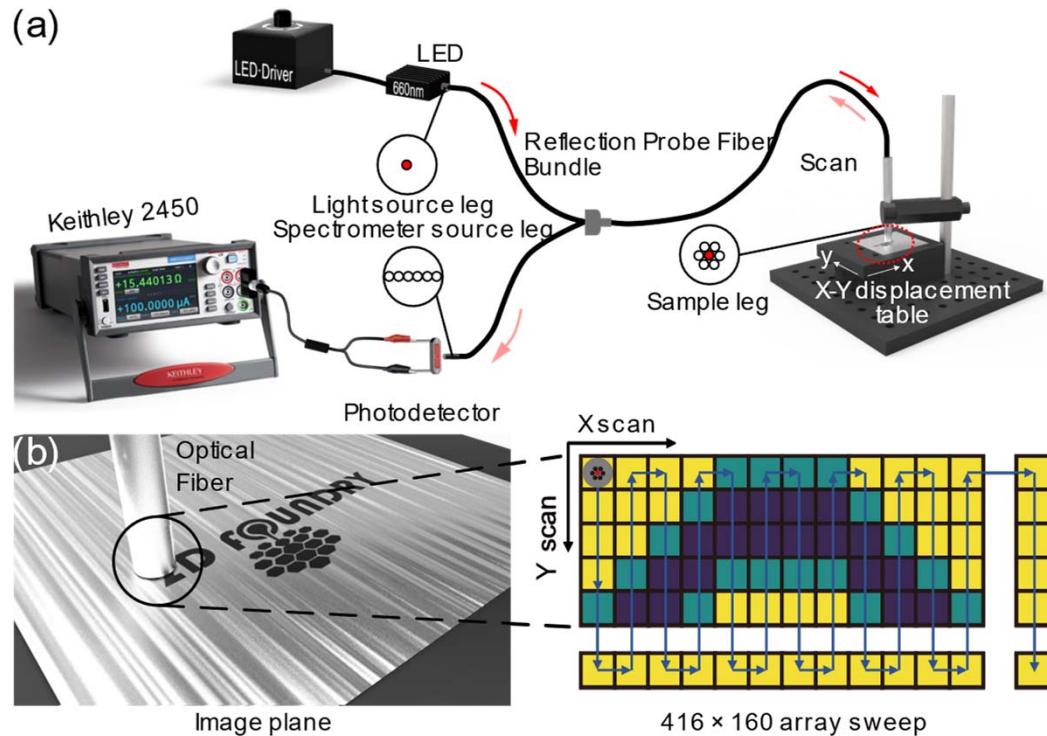
b4

Prompt 4-input

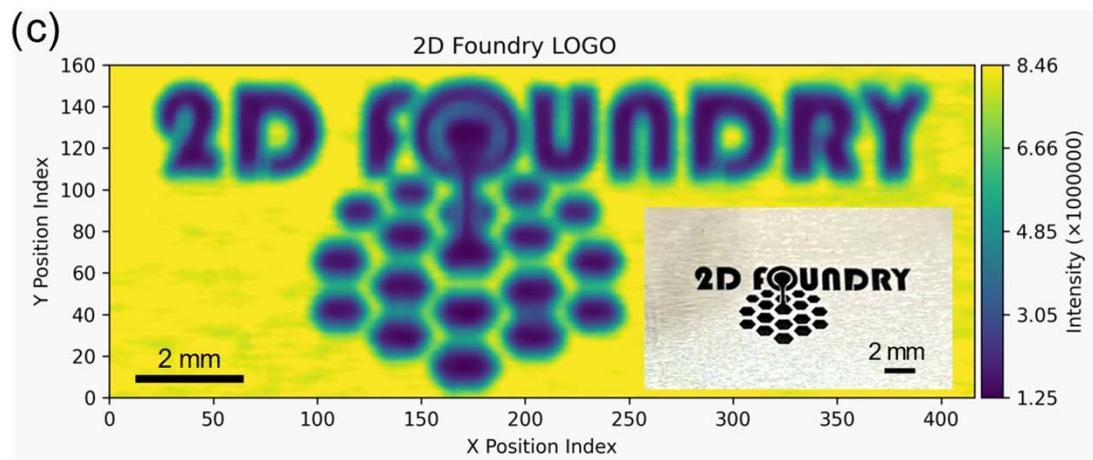
Save the datasets with the timestamp. Can you write from the **very basic, simple code, and step by step, stop at the every step for me to confirm it works (and give me the feedback of it is successfully if it works, then can process to the next step) to reach the goal here for me?**

The key tricks: Divide the problems into small units
Step by step feedback loop

Towards autonomous laboratory instrumentation control with LLM-based tools



Single pixel camera



Towards autonomous laboratory instrumentation control with LLM-based tools

(a)

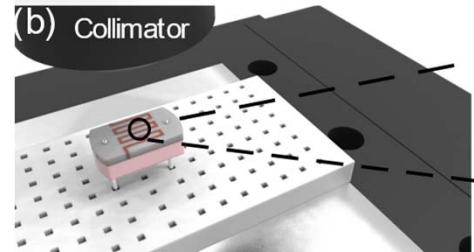
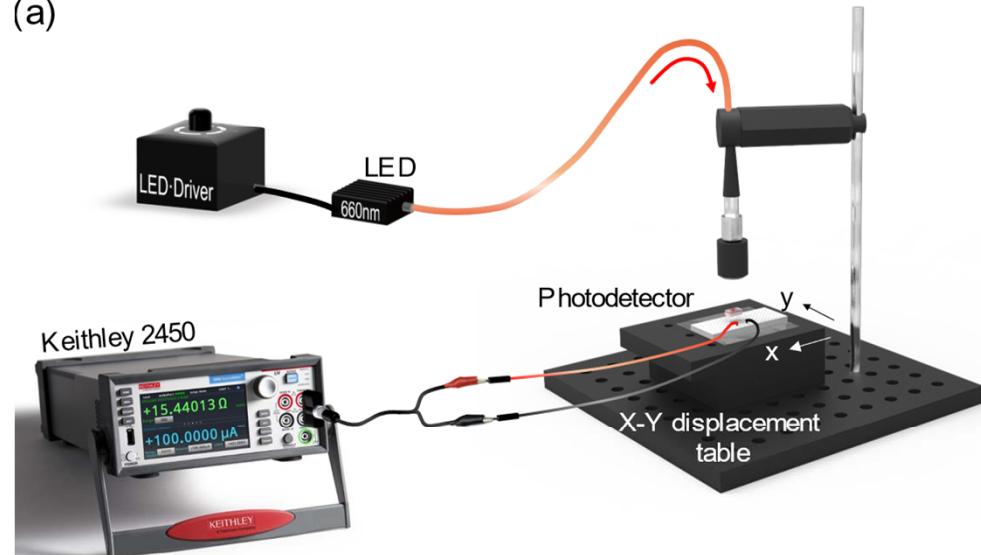
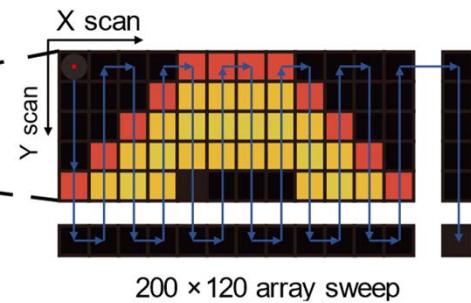
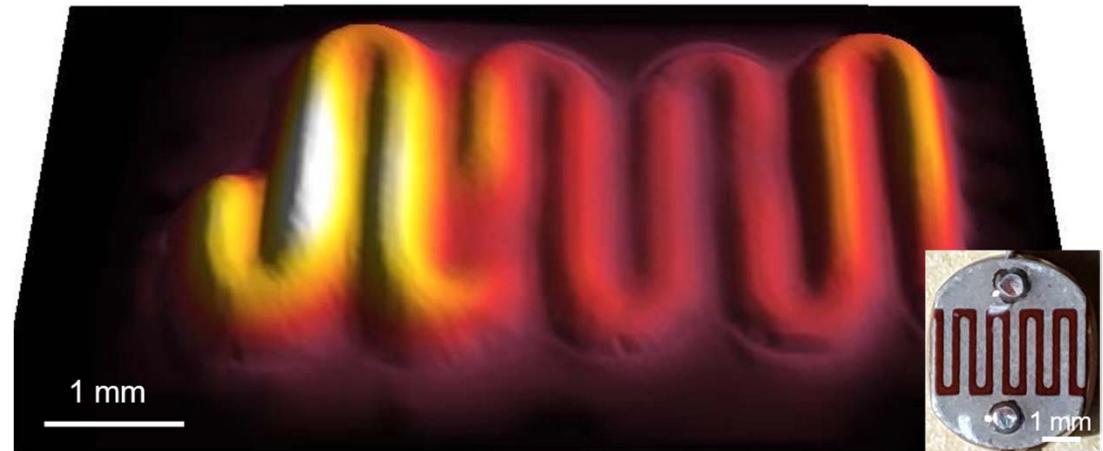


Image plane



Scanning photocurrent mapping

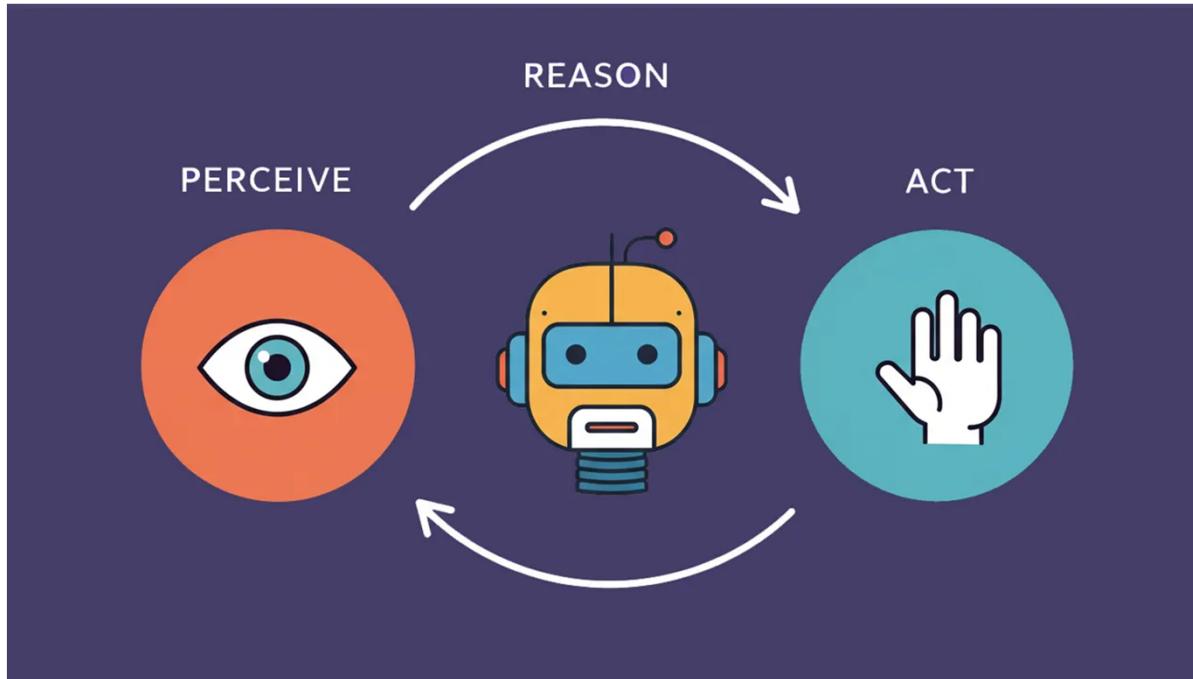


Small Struct. 6, 2500173 (2025)

We were told that 2025 would be
the year of agents.

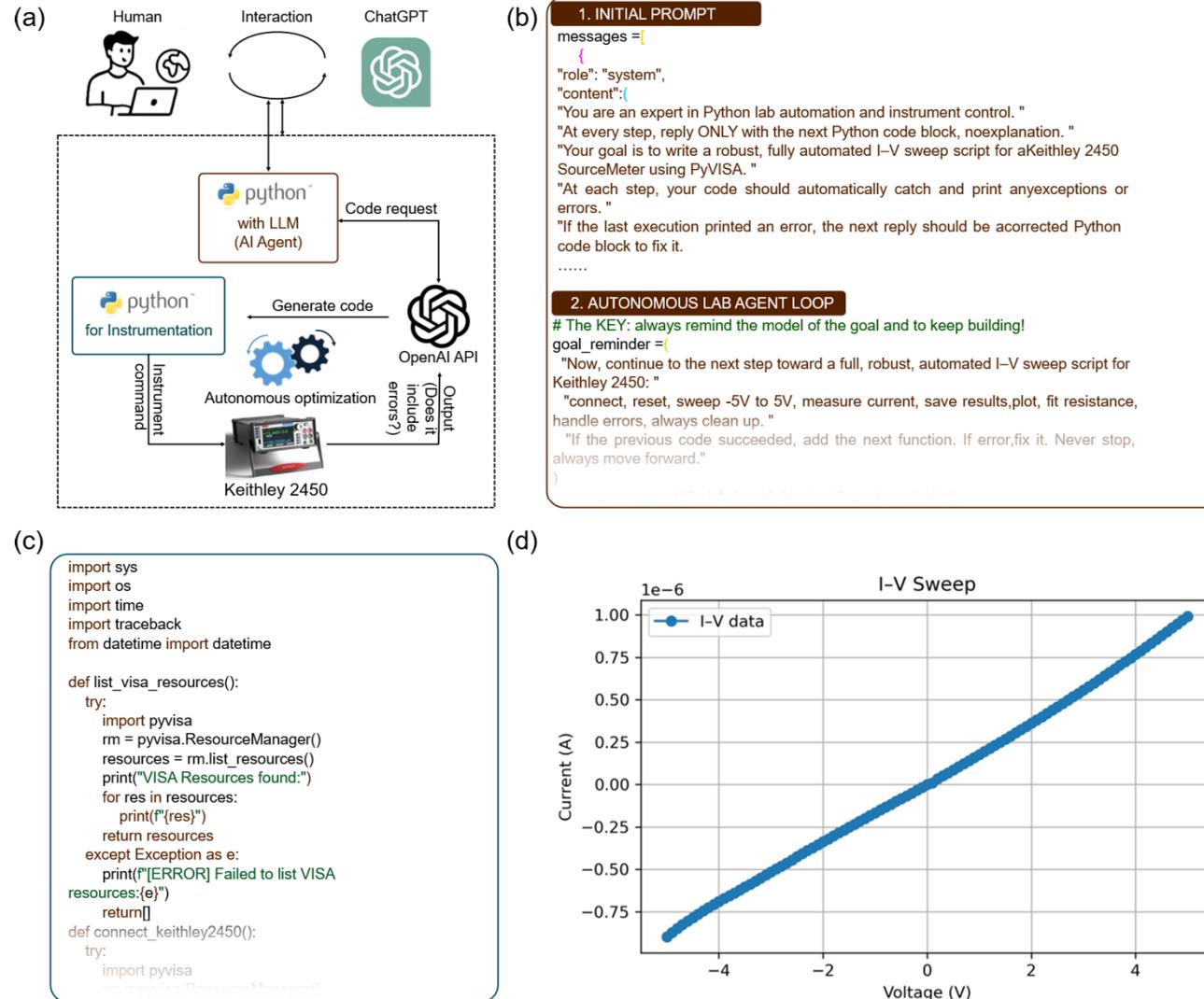


AI Agents



Hello AI Agents: Goodbye UI Design, RIP Accessibility from [Jakob Nielsen](#)

Towards autonomous laboratory instrumentation control with LLM-based tools



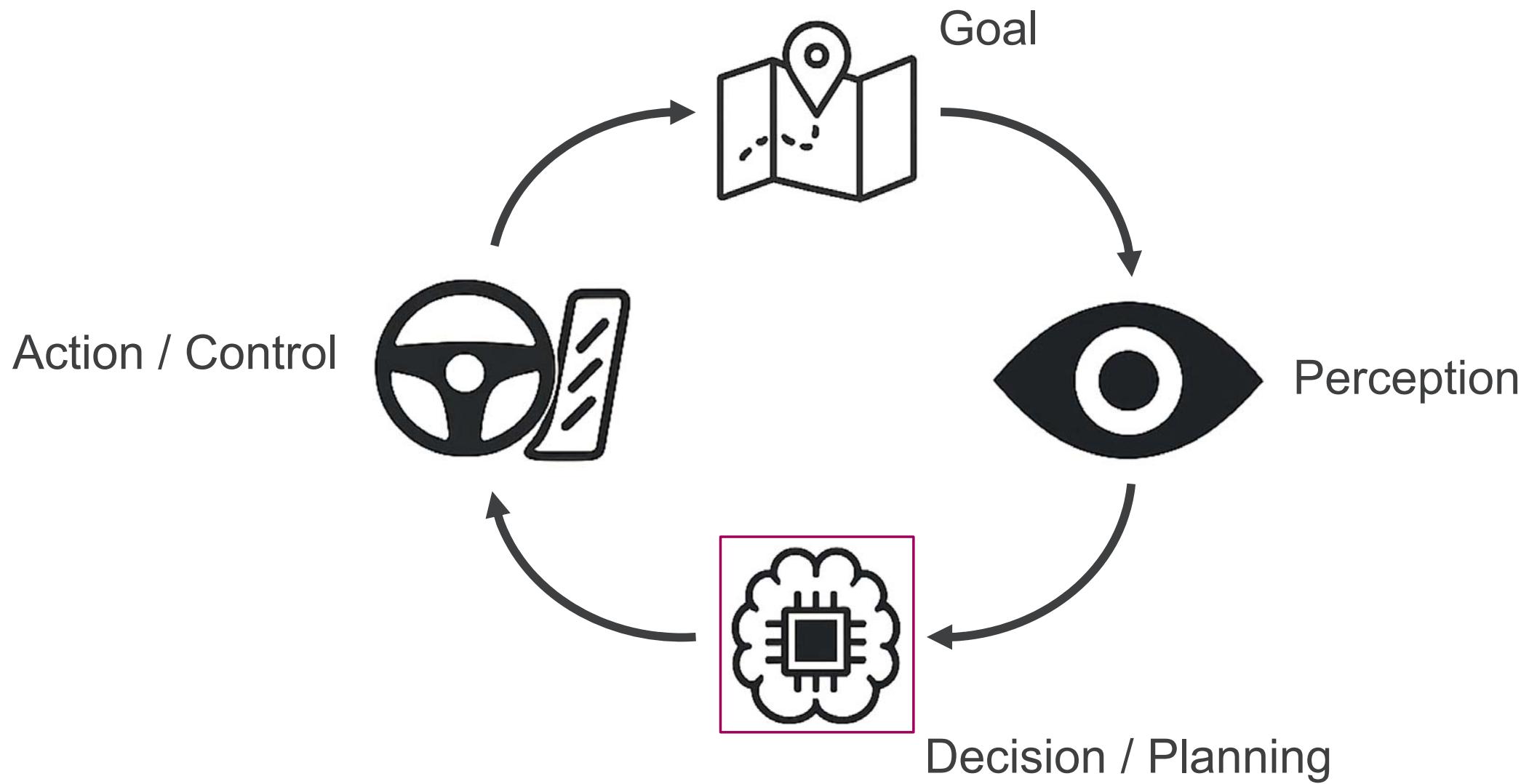
The demonstration of the AI agent for scientific instrumentation

The screenshot shows the Spyder Python IDE interface. The left sidebar displays a file tree with several folders and files, including '.env', 'autonomous_keithley_lab.py', and 'record_temp'. The main editor window contains a Python script named 'autonomous_keithley_lab.py'. The script uses the OpenAI API to interact with a Keithley 2450 SourceMeter. It starts by importing io, sys, traceback, dotenv, and openai. It then loads environment variables and initializes an OpenAI client. The script defines an 'INITIAL PROMPT' message for the AI agent, which provides instructions for writing a robust, fully automated I-V sweep script. This includes handling errors, auto-detecting the Keithley 2450 on the VISA bus, and safely configuring it. The AI is also asked to sweep voltage from -5V to +5V, measure current, save data, and handle errors gracefully. The script then enters a loop where it sends messages to the AI, receives completions, and extracts the generated Python code to execute. The right side of the interface shows the 'Usage' help panel and a Python console window.

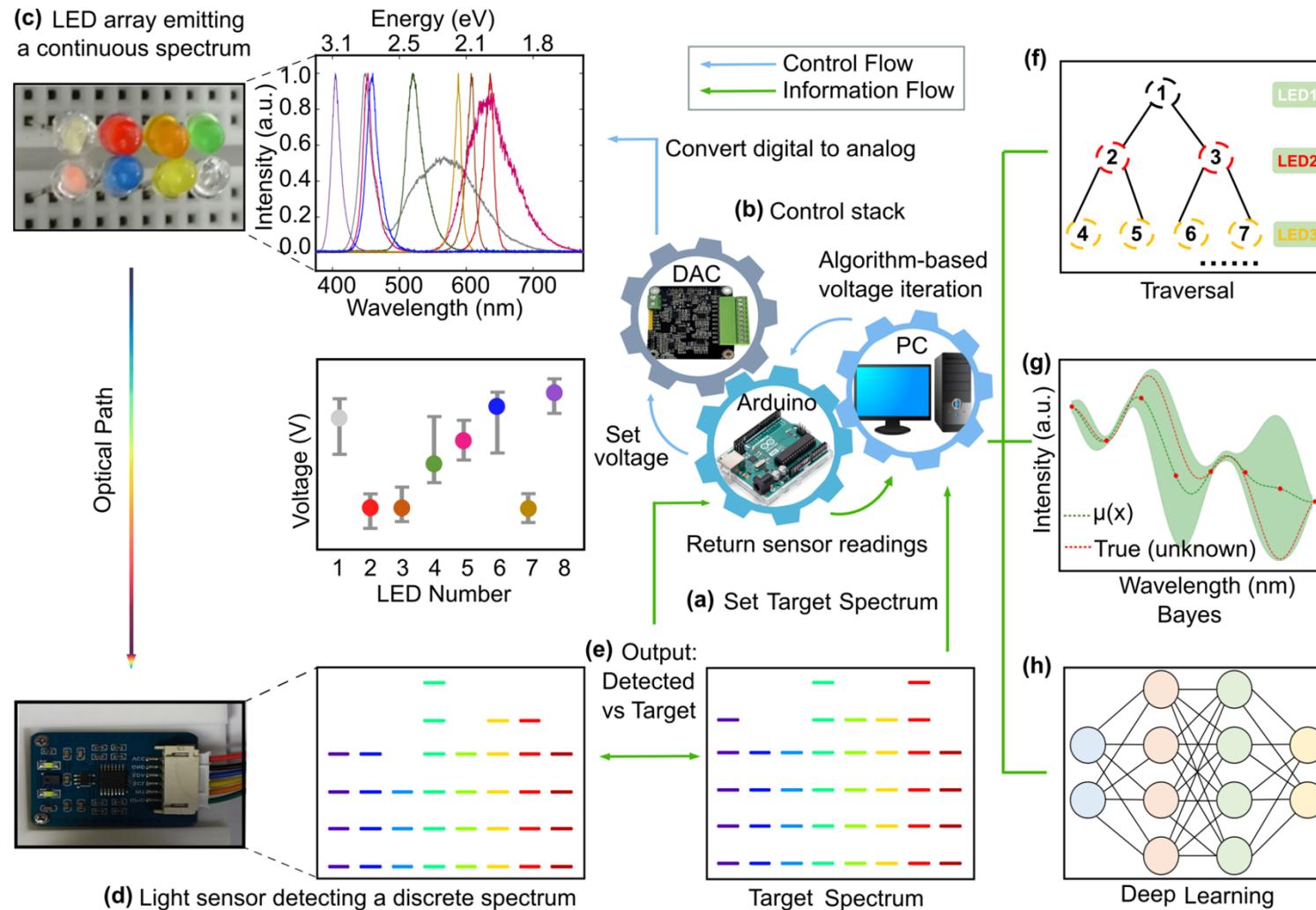
```
import io
import sys
import traceback
from dotenv import load_dotenv
from openai import openAI
# 0. SETUP
load_dotenv()
client = OpenAI()
# 1. INITIAL PROMPT
messages = [
    {
        "role": "system",
        "content": (
            "You are an expert in Python lab automation and instrument control.\n"
            "At every step, reply ONLY with the next Python code block, no explanation.\n"
            "Your goal is to write a robust, fully automated I-V sweep script for a Keithley 2450 SourceMeter.\n"
            "At each step, your code should automatically catch and print any exceptions or errors.\n"
            "If an error occurs during execution, the next step should be a corrected Python code block.\n"
            "Repeat this process, automatically debugging and improving the code, until the script runs without any errors.\n"
            "The final script should:\n"
            "• Auto-detect the Keithley 2450 on the VISA bus.\n"
            "• Configure it safely.\n"
            "• Sweep voltage from -5V to +5V, measure current, save and plot.\n"
            "• Handle errors gracefully and always clean up."
        )
    },
    {
        "role": "user",
        "content": (
            "Start by sending Python code to list all VISA resources.\n"
            "If any error occurs during execution, the next step should automatically fix the code based\n            \"without waiting for user input. Repeat this process until the full I-V sweep is successful.\"
        )
    }
]
# 2. AUTONOMOUS LAB AGENT LOOP
for iteration in range(20): # Up to 30 cycles (should finish in <15)
    print(f"\n--- Iteration {iteration+1} ---\n")
    resp = client.chat.completions.create(
        model="o3",
        messages=messages,
        reasoning_effort='high',
        max_completion_tokens=2048
    )
    code = resp.choices[0].message.content
    # Extract code (remove code fences)
    if code.startswith('```python'):
        code = code.split('```python')[1].split('```')[0].strip()
    elif code.startswith('```'):
        code = code.split('```')[1].split('```')[0].strip()
```

When we run the AI Agent code in Python…

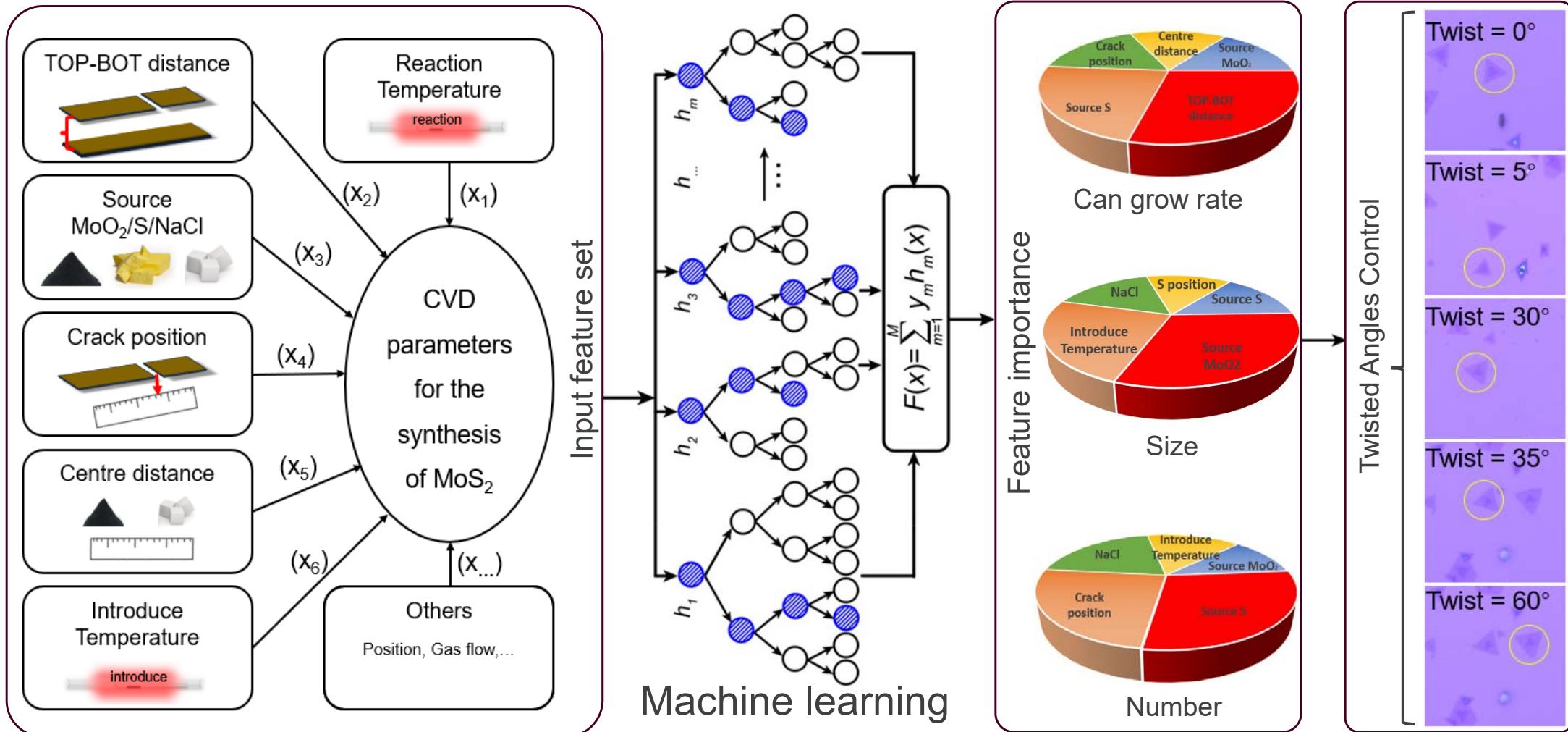
The concept of autonomous (self-driving) lab



Towards autonomous laboratory



Towards autonomous synthesis for CVD 2D materials

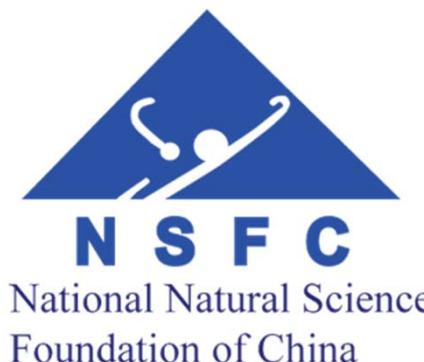


Acknowledgement



European Research Council

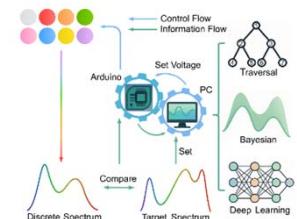
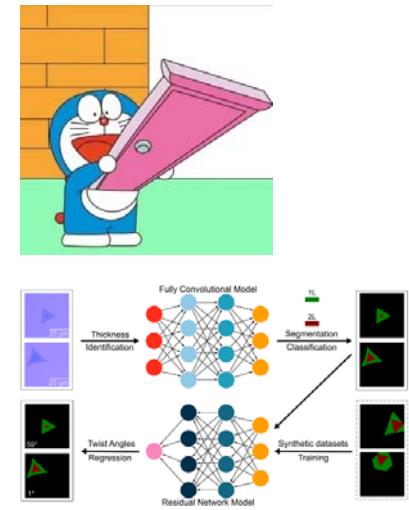
- ICMM: 2D Foundry group
- ICMM: Dr. Eduardo R. Hernández



- CAS: Prof. Pingheng Tan
Dr. Yan Zhou
- Students from Xidian University:
Q. Lei, H. Yang, X. Chen, K. He...

Take home message

- Deep learning enables accurate recognition of twist angles in CVD-grown 2D materials
- LLM-powered tools can autonomously control laboratory instrumentation
- Autonomous lab systems could accelerate materials discovery



Thanks for your attention!