Towards Full Autonomous Synthesis and Characterization of 2D Materials

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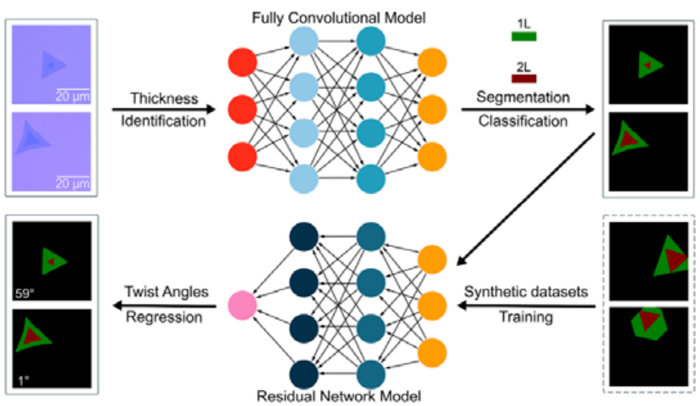
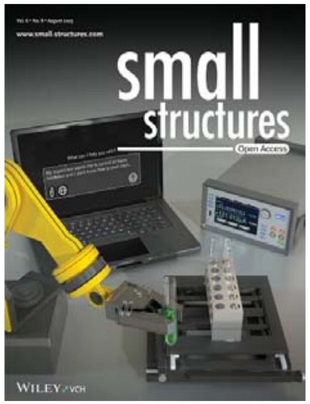
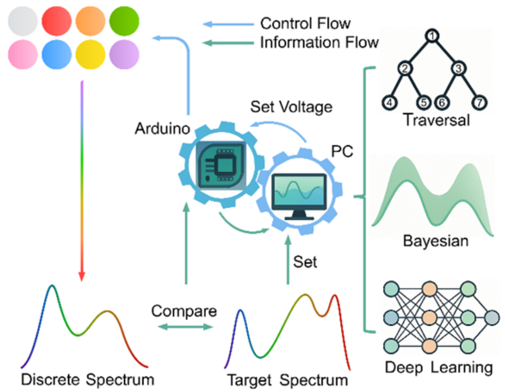






- **Motivation**

- **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

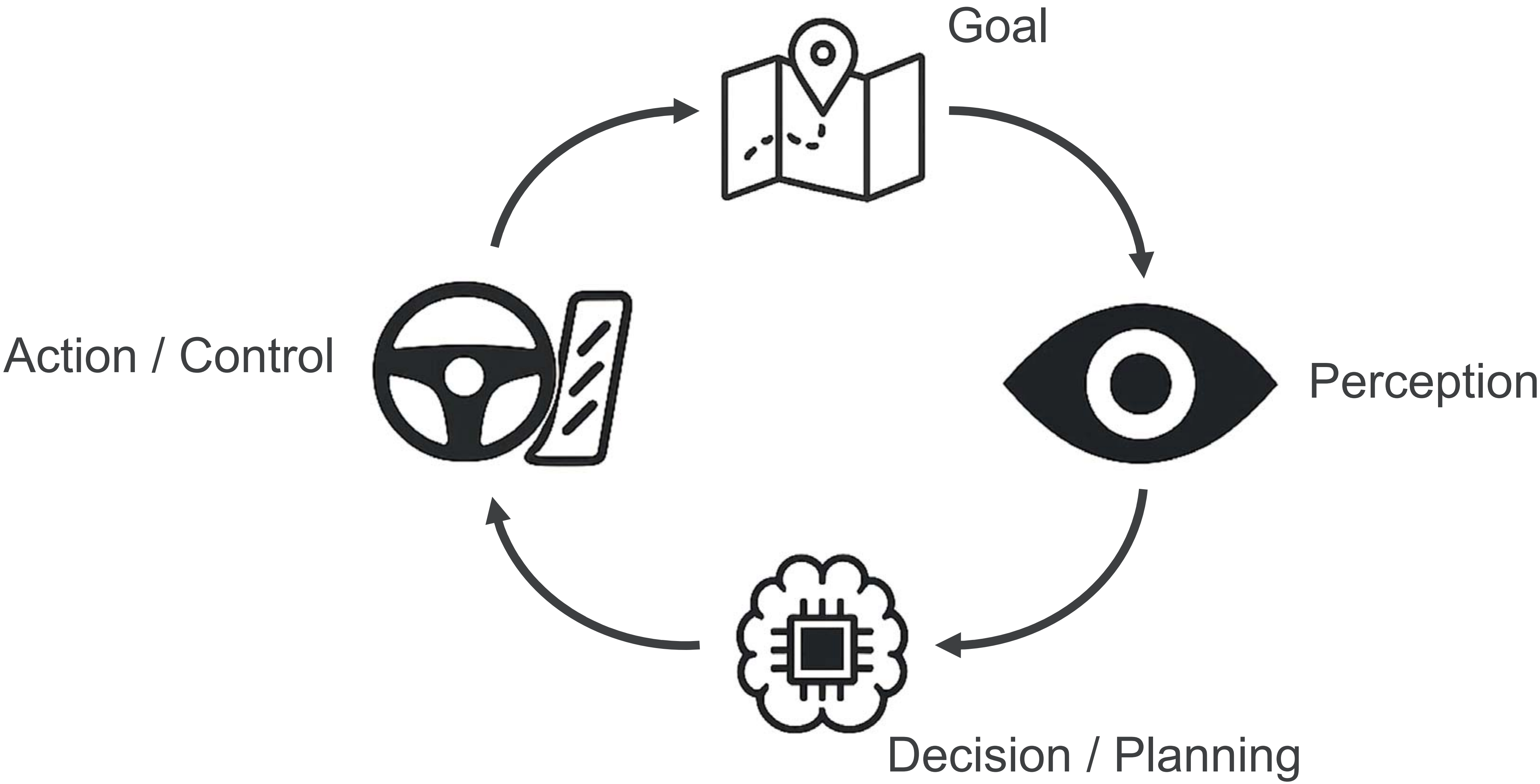
Can we make everything autonomously?

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**.

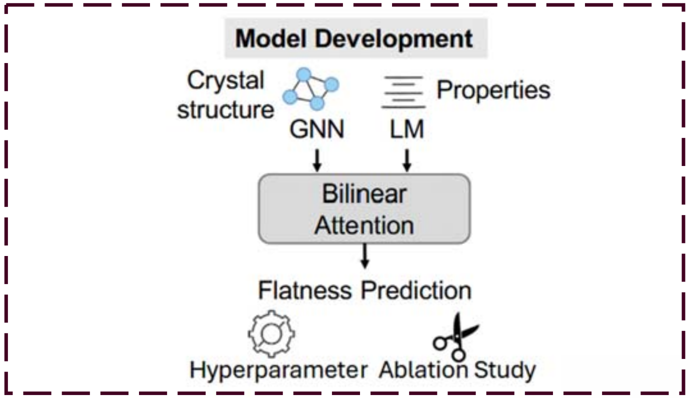
Doraemon (ドラえもん) is a Japanese manga series written and illustrated by Fujiko F. Fujio

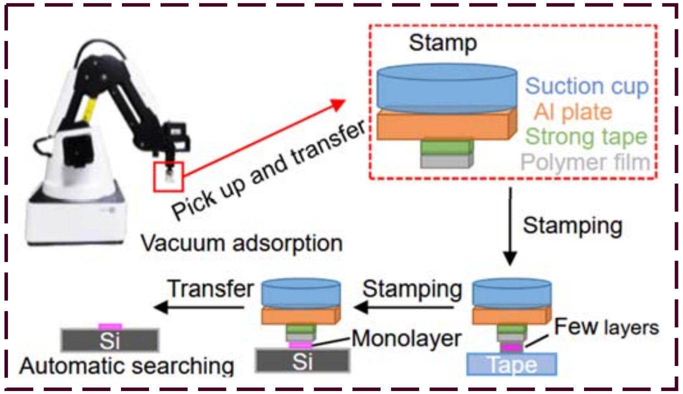
The concept of autonomous (self-driving) lab



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





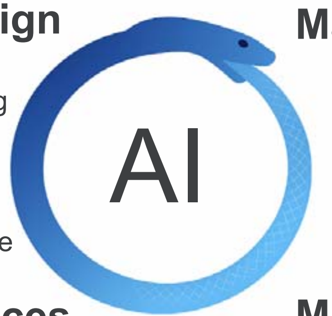
K. Matsuda *et. al*, Autonomous robotic mechanical exfoliation of two-dimensional semiconductors combined with Bayesian optimization *arXiv*: 2411.06891v1

Design

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

Materials Synthesis

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



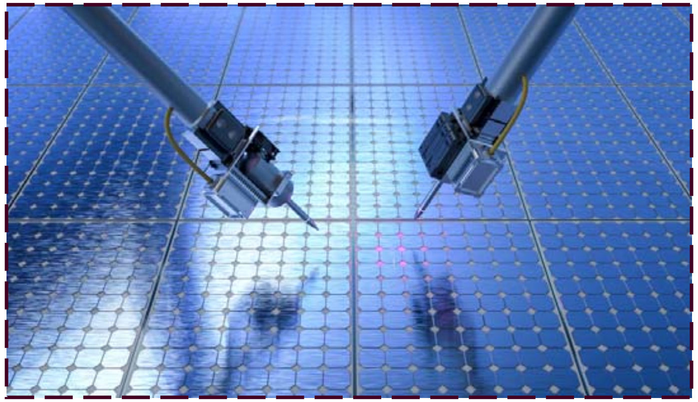
Robotic assembly and **automated** probing accelerate device prototyping, ensuring precise stacking and scalable integration.

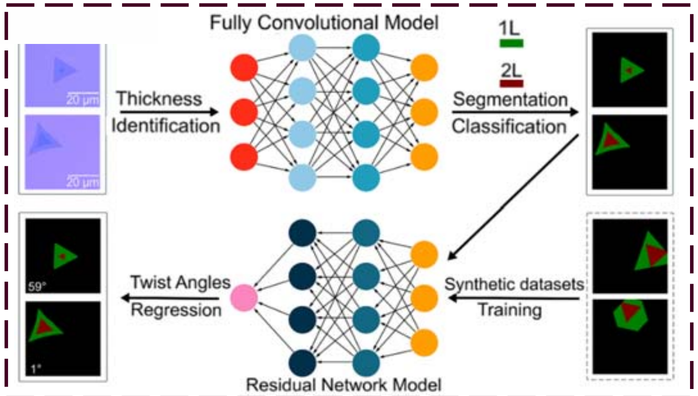
Devices

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

Materials Characterization

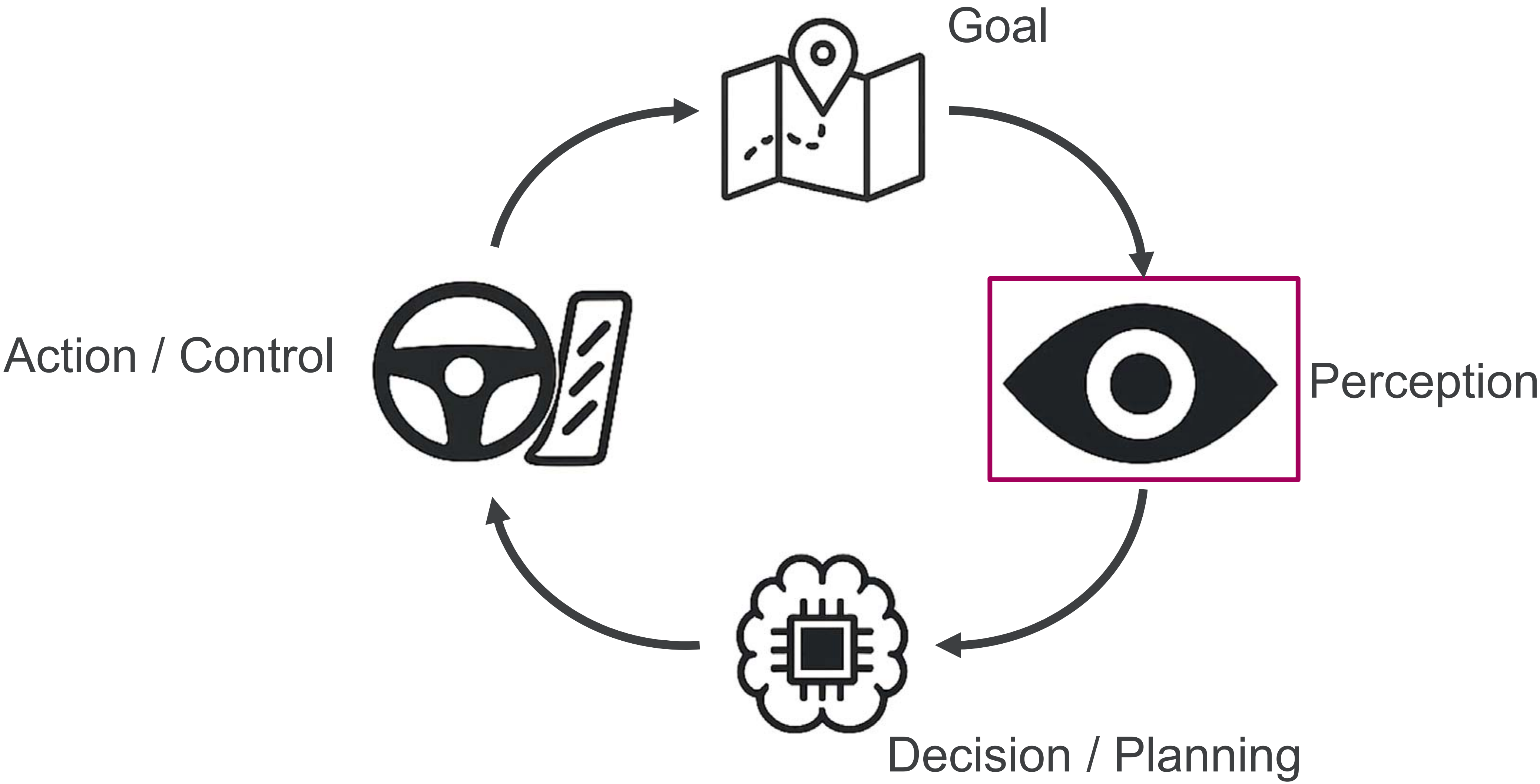
T. Buonassisi *et. al*, A self-supervised robotic system for autonomous contact-based spatial mapping of semiconductor properties *Sci. Adv.* 11, eadw7071 (2025)





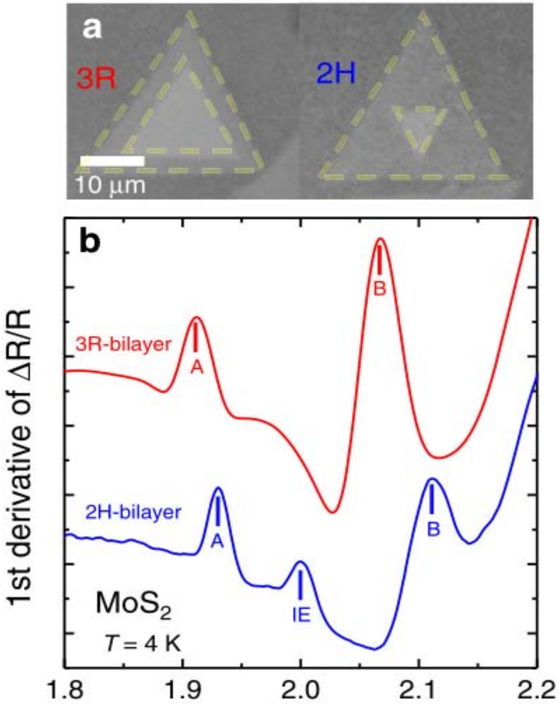
Y. Xie *et. al*, Identification and structural characterization of twisted atomically thin bilayer materials by deep learning *Nano Lett.* 24 (9), 2789-2797 (2024)

The concept of autonomous (self-driving) lab

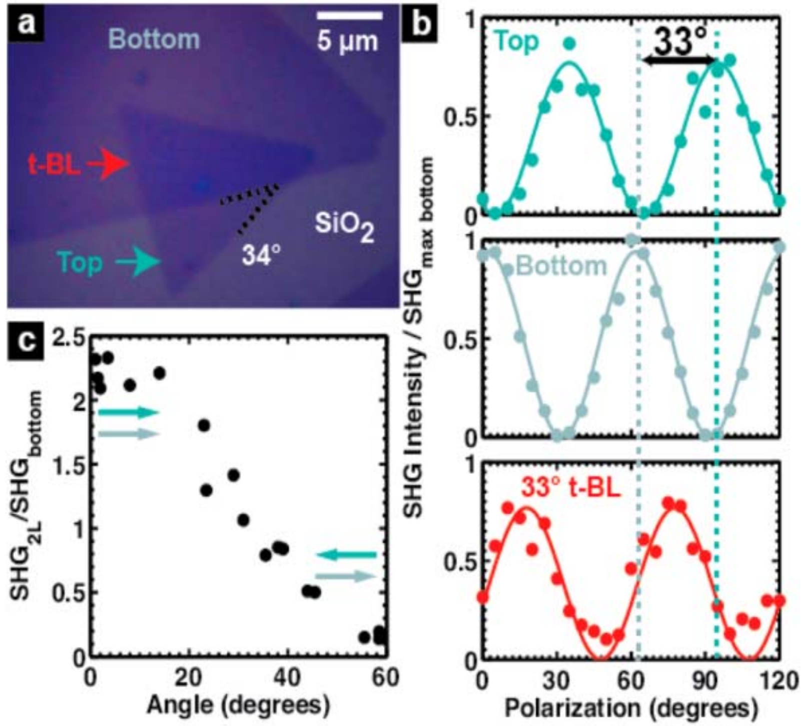


Motivation

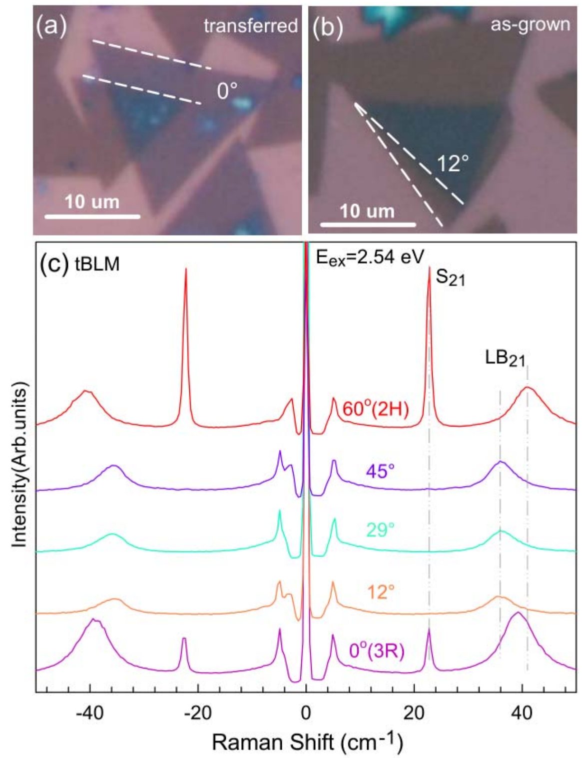
The current measurement methods for twist angles in bilayer TMDs



Differential Reflectance [1]



Second-harmonic generation [2]



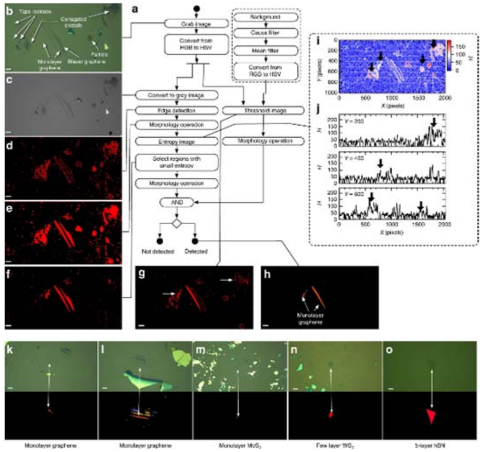
Low wavenumber Raman spectra [3]

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

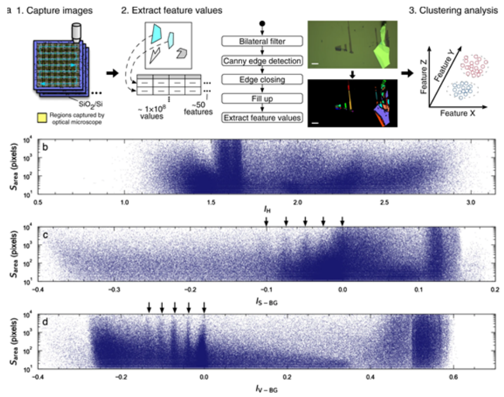
[2] *Nano Lett. 2014 Jul 9;14(7):3869-75.*

[3] *ACS Nano 2018, 12, 8, 8770–8780*

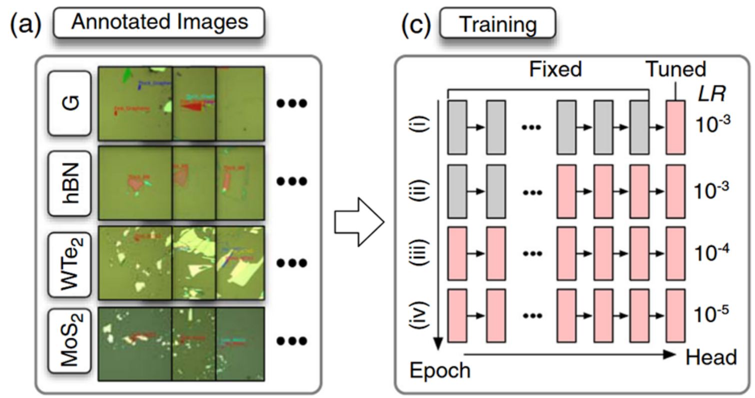
Motivation



Nat Commun **9**, 1413 (2018)

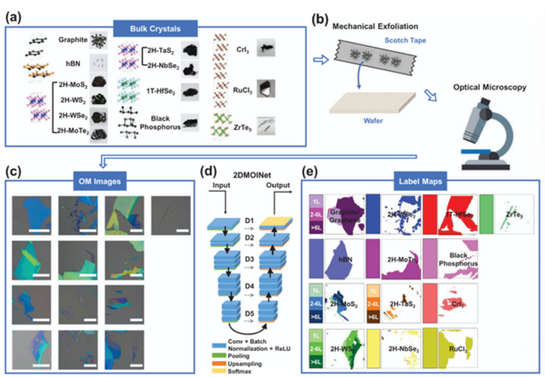


npj 2D Mater Appl **3, 4** (2019).

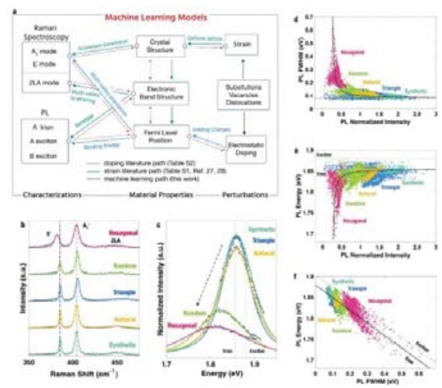


npj 2D Mater Appl **4**, 3 (2020).

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



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

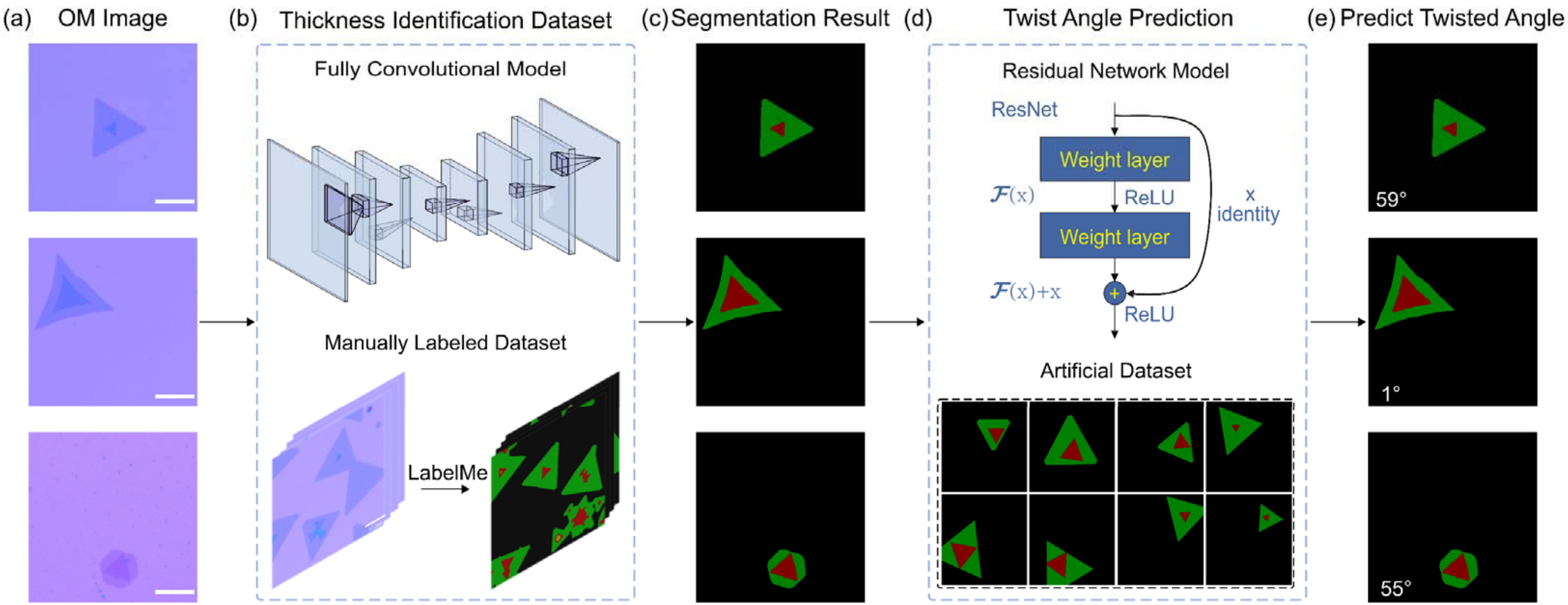


The identification of contrast, color, edges, shapes, flake sizes, and their distributions is crucial for advancing research on 2D materials and their device applications.

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



Thickness

Classification Task

Twisted Angles

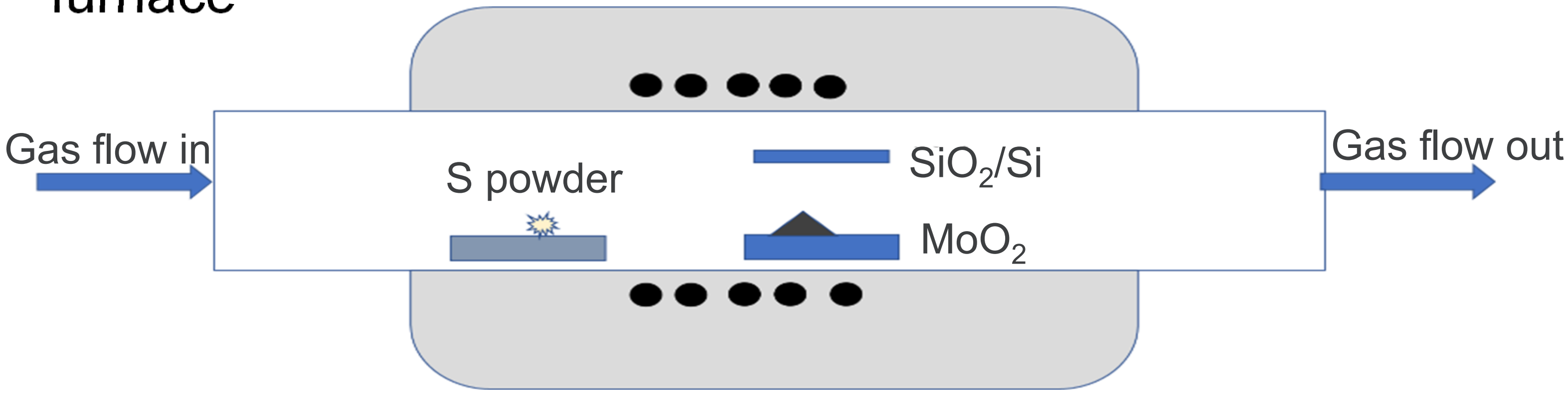
Regression Task

Data Preparation （CVD growth process）

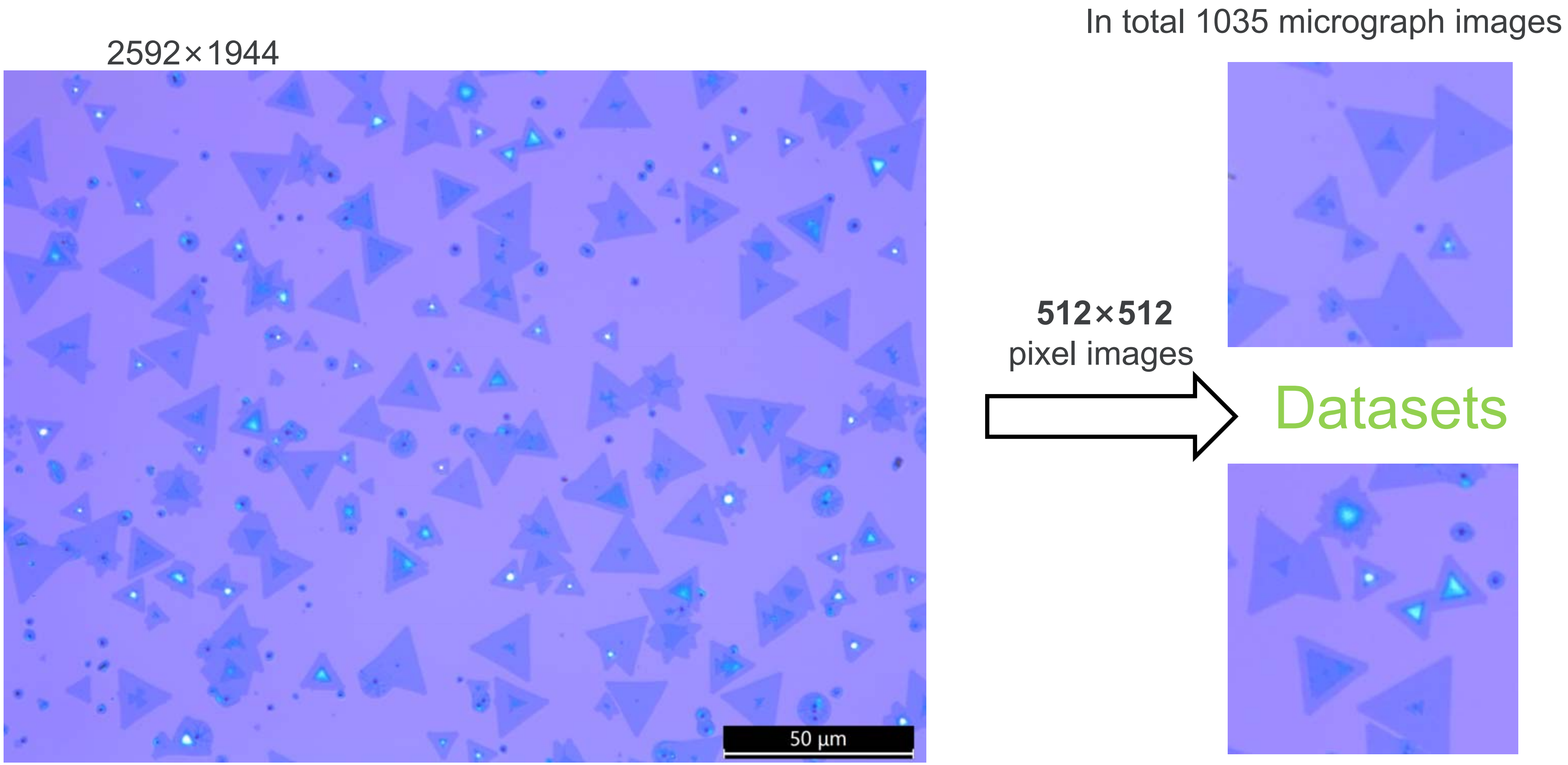
Thermo Scientific
Single-zone tube furnace

Alicat Scientific
Gas flow control valve

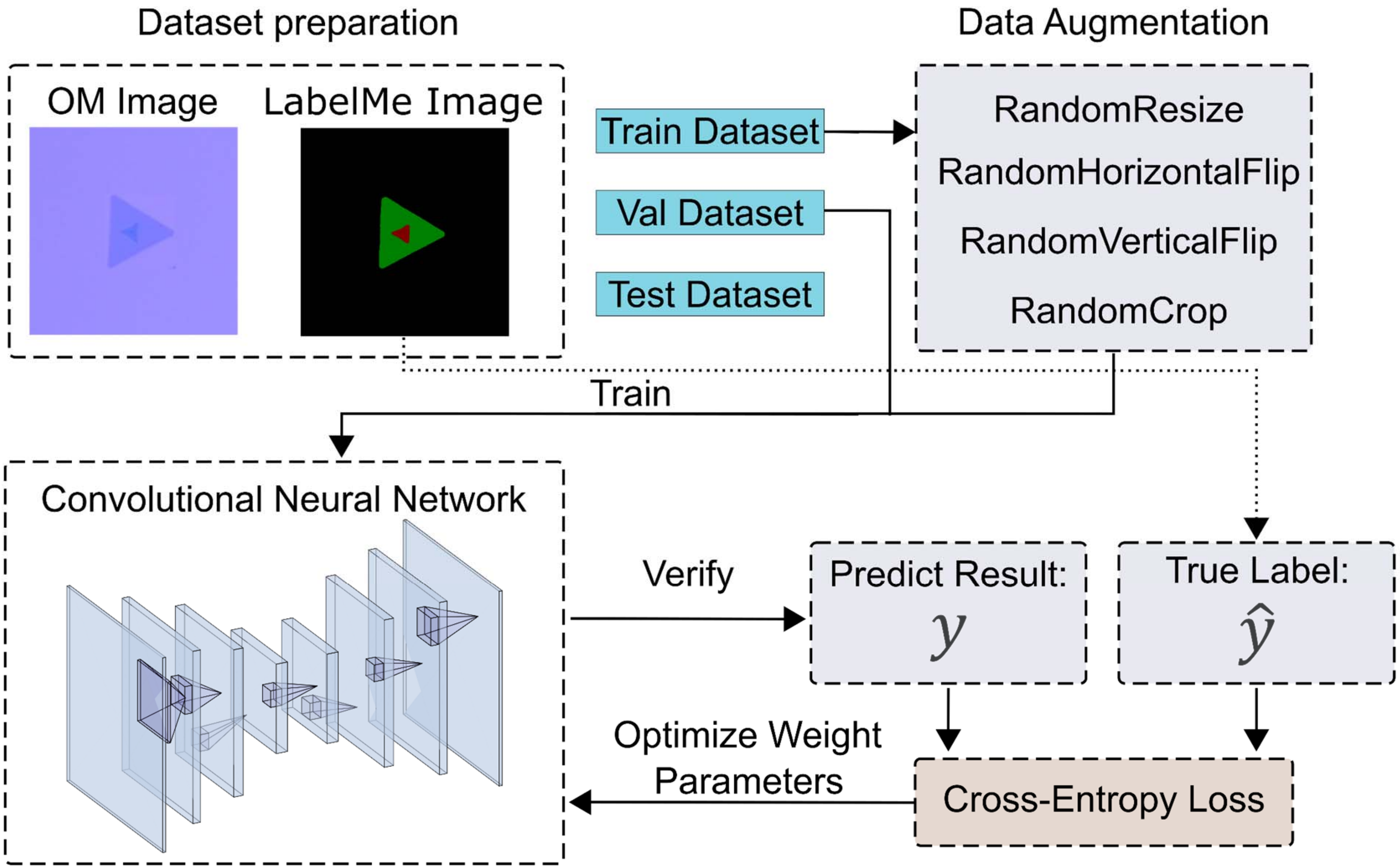
Ar Supply



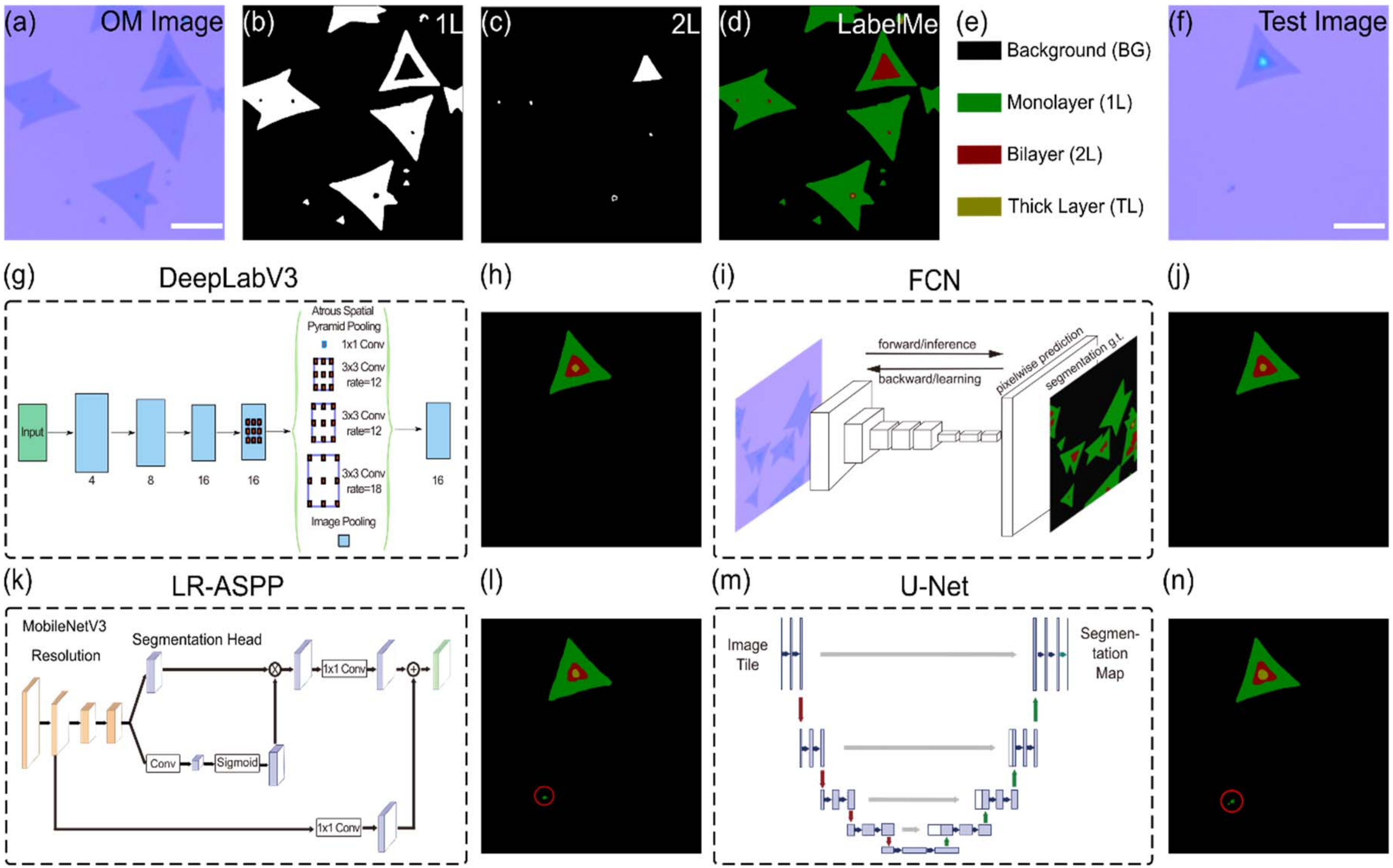
Data preprocessing



Deep Learning to Identify the Thickness of TMDs

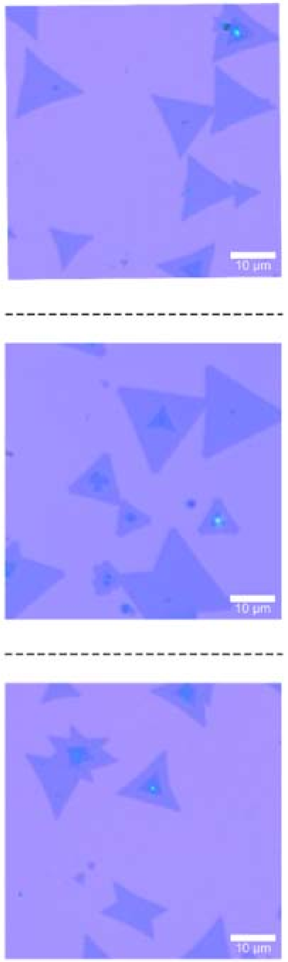


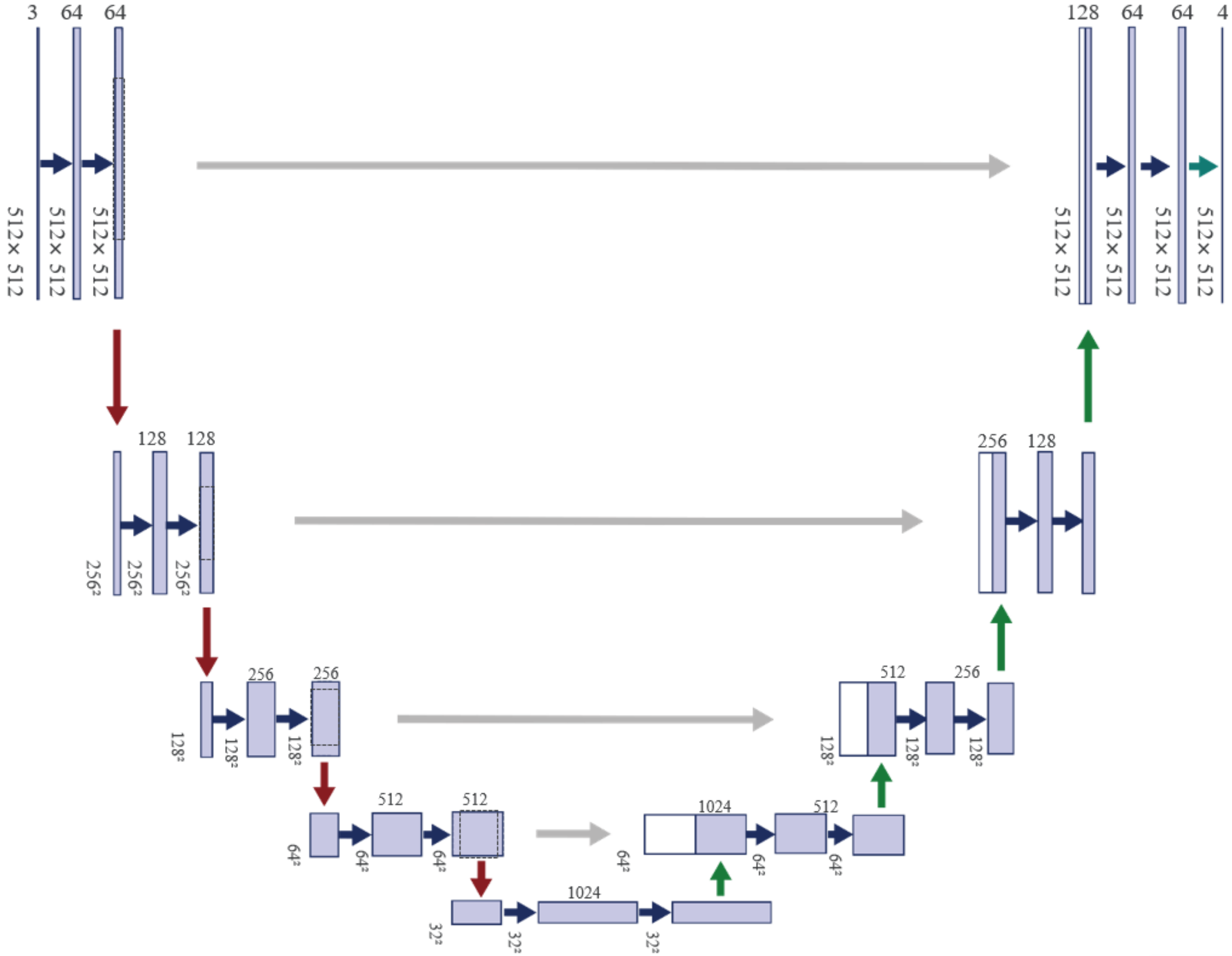
Deep Learning to Identify the Thickness of TMDs



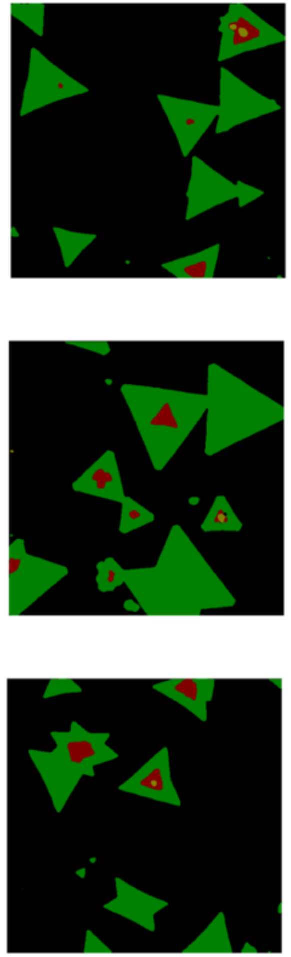
Deep Learning to Identify the Thickness of TMDs

Input Images





Output Images



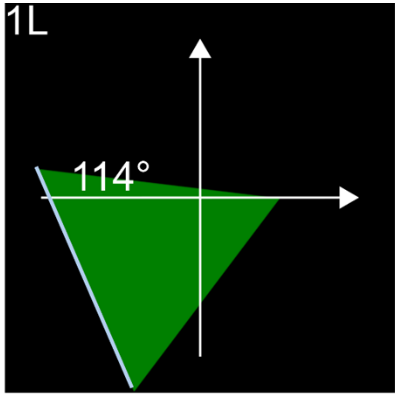
Ronneberger O et al., Medical Image Computing and Computer-Assisted Intervention–MICCAI 2015: 18th International Conference, 234-241, 2015.

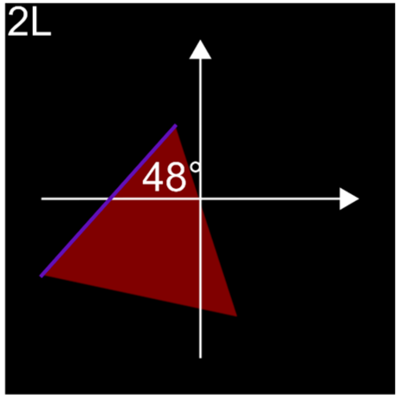
Deep Learning to identify Twist Angles

1.Find the leftmost side of each triangle.

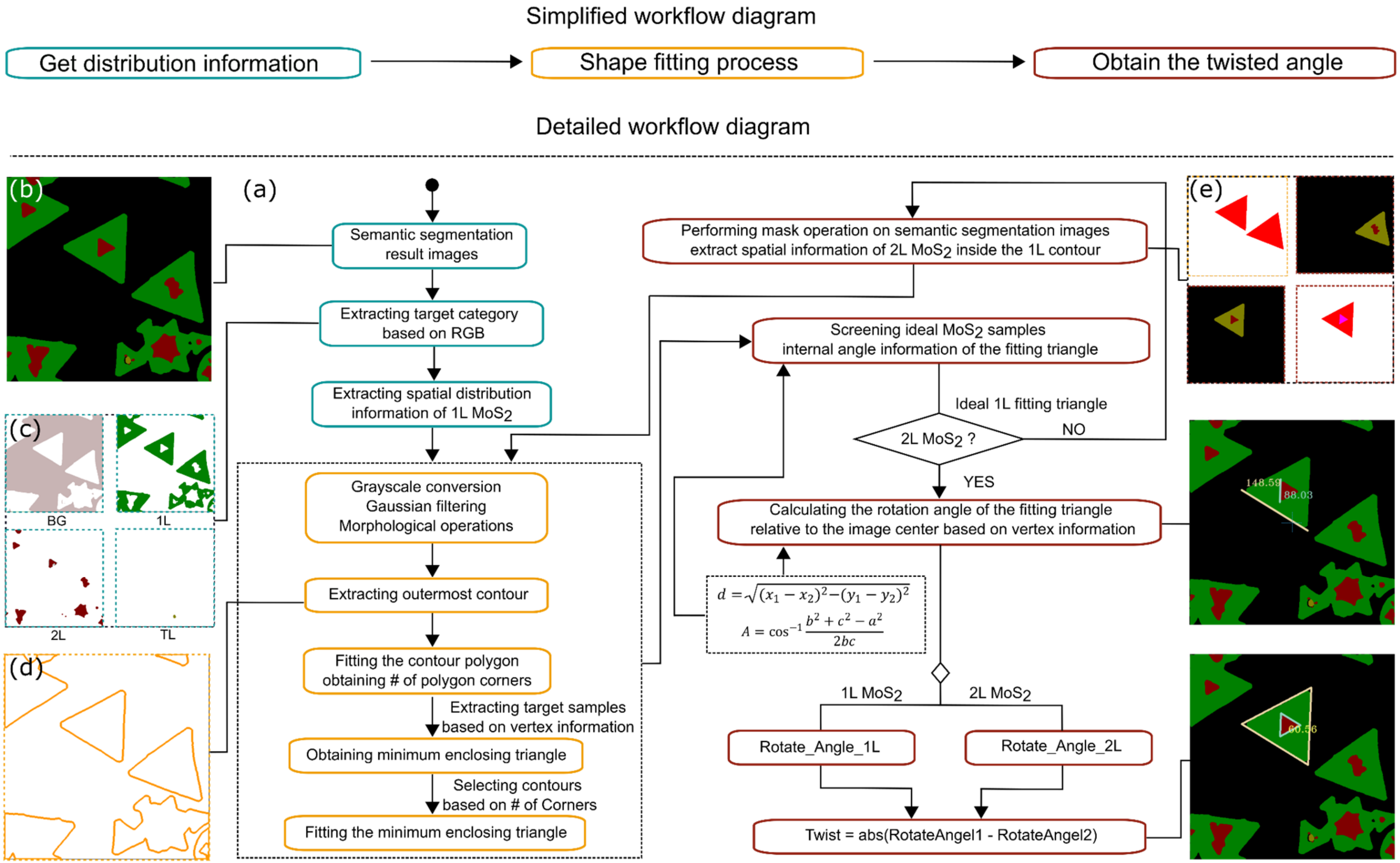
2.Determine its rotation angle relative to the center of the image

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

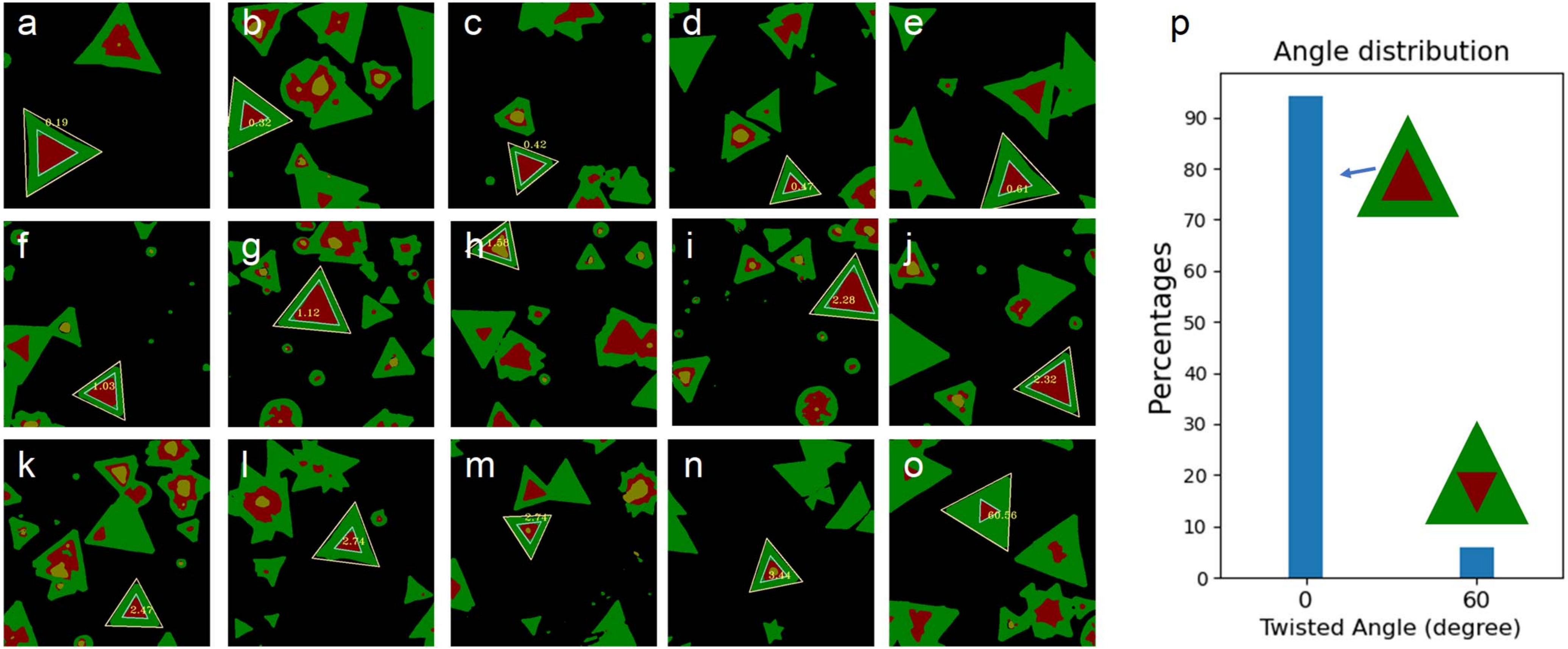




OpenCV to Identify Twisted Angles of TMDs



OpenCV to Predict the Twisted Angles of TMDs



Half intelligence

MIT News
ON CAMPUS AND AROUND THE WORLD
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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?

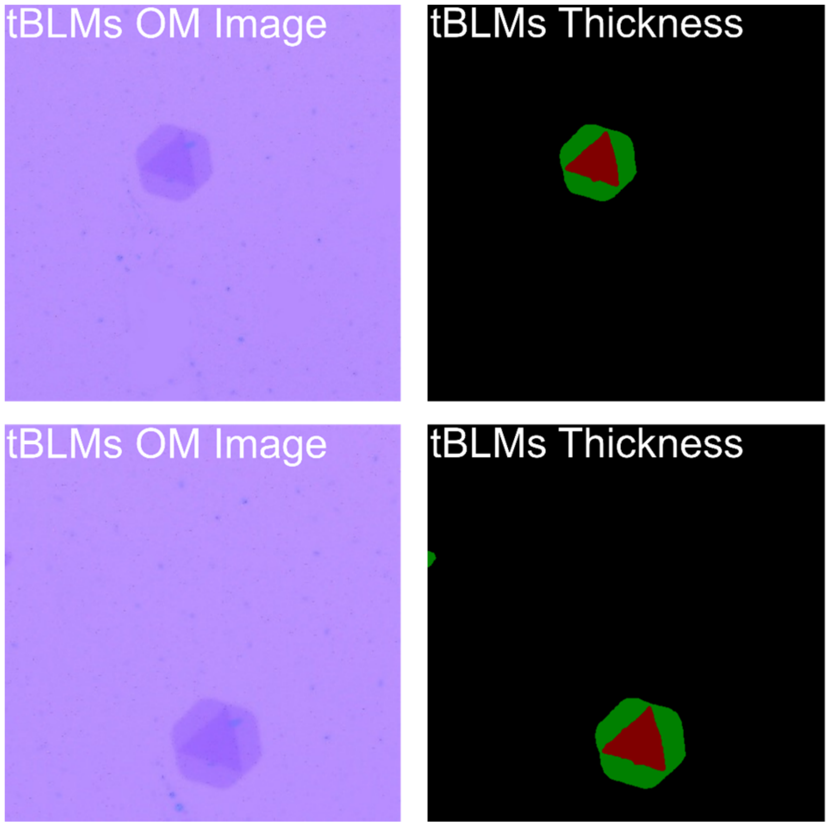
↓

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

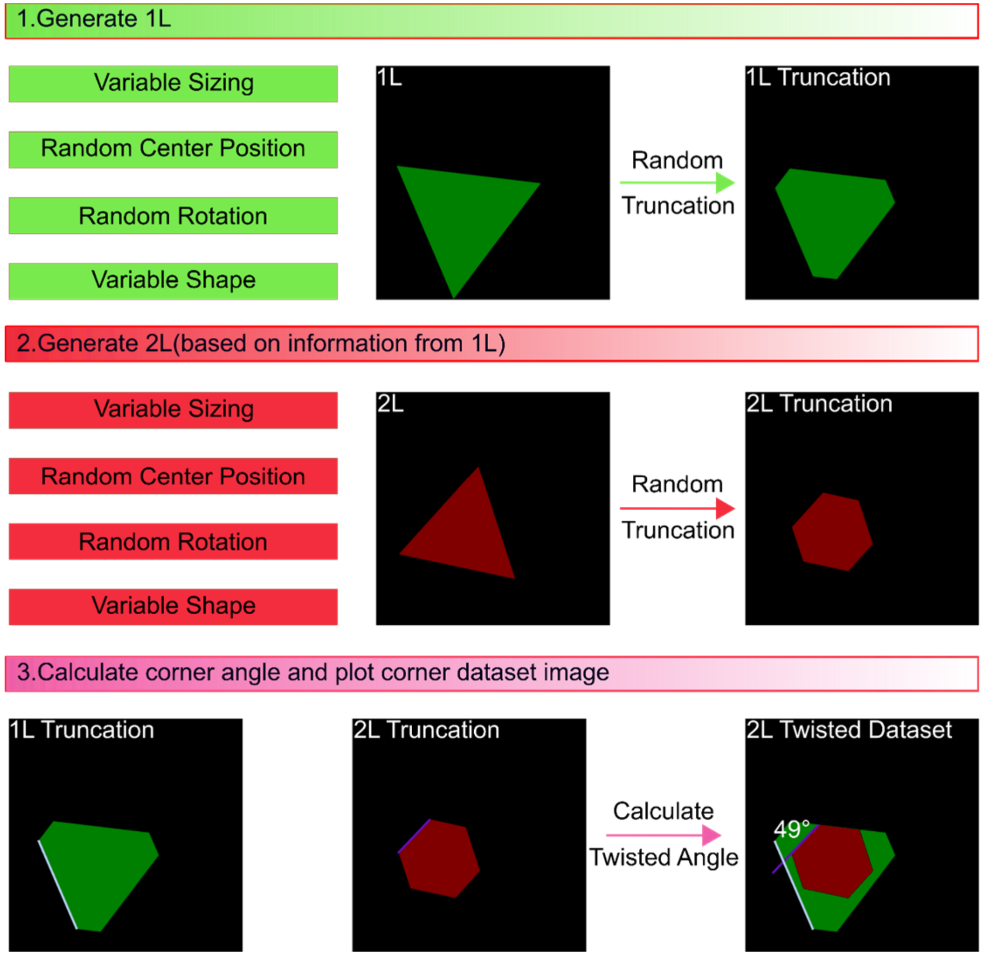
https://news.mit.edu/2020/mit-toyota-release-visual-open-data-accelerate-autonomous-driving-research-0618

Training Dataset Preparation

True Datasets



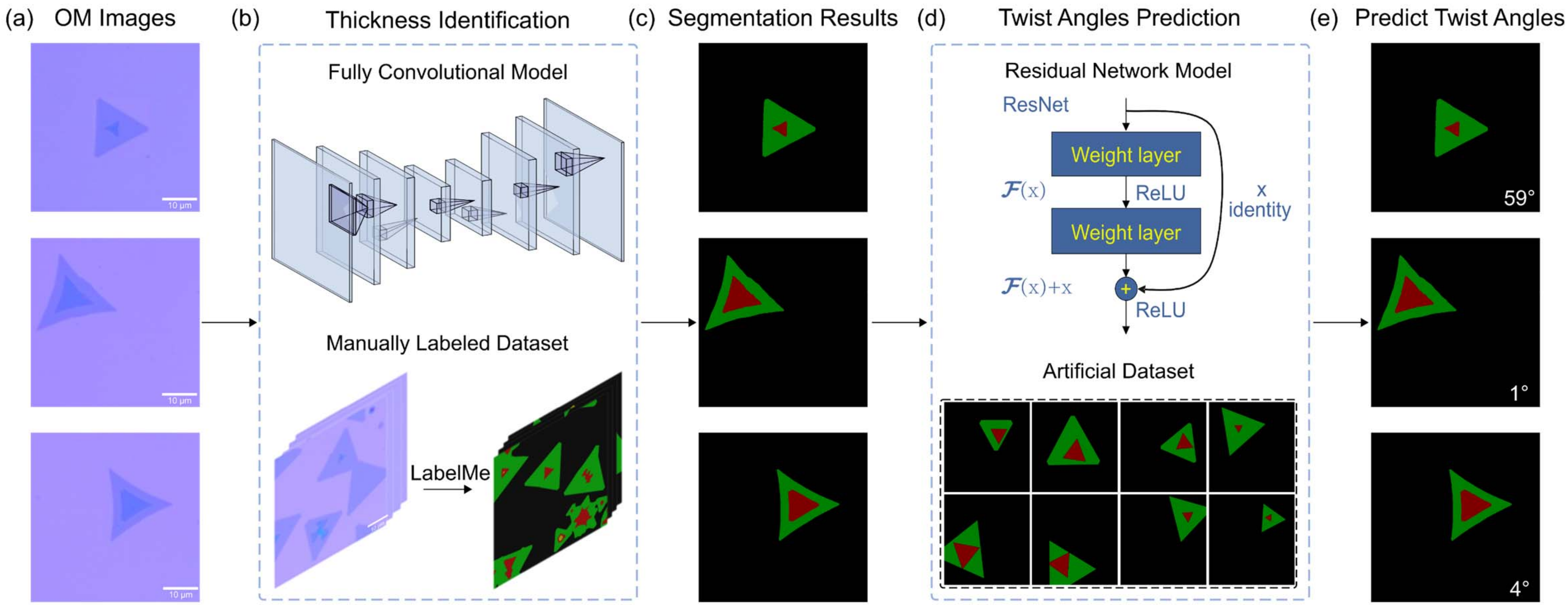
Artificial Datasets



Eduardo R. Hernandez

Nano Lett. , 24, 9, 2789–2797 (2024)

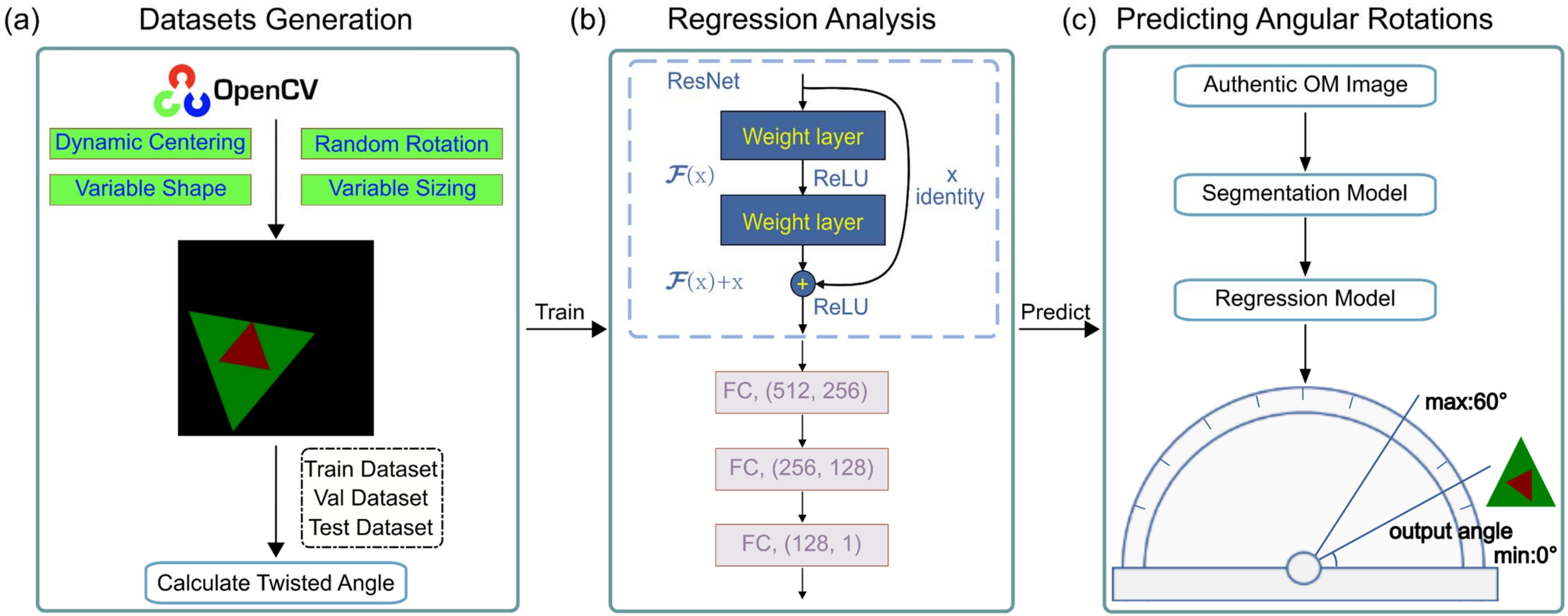
Deep Learning to Predict Twist Angles



Full intelligence

Nano Lett., 24, 9, 2789–2797 (2024)

Deep Learning Predict Twist Angles



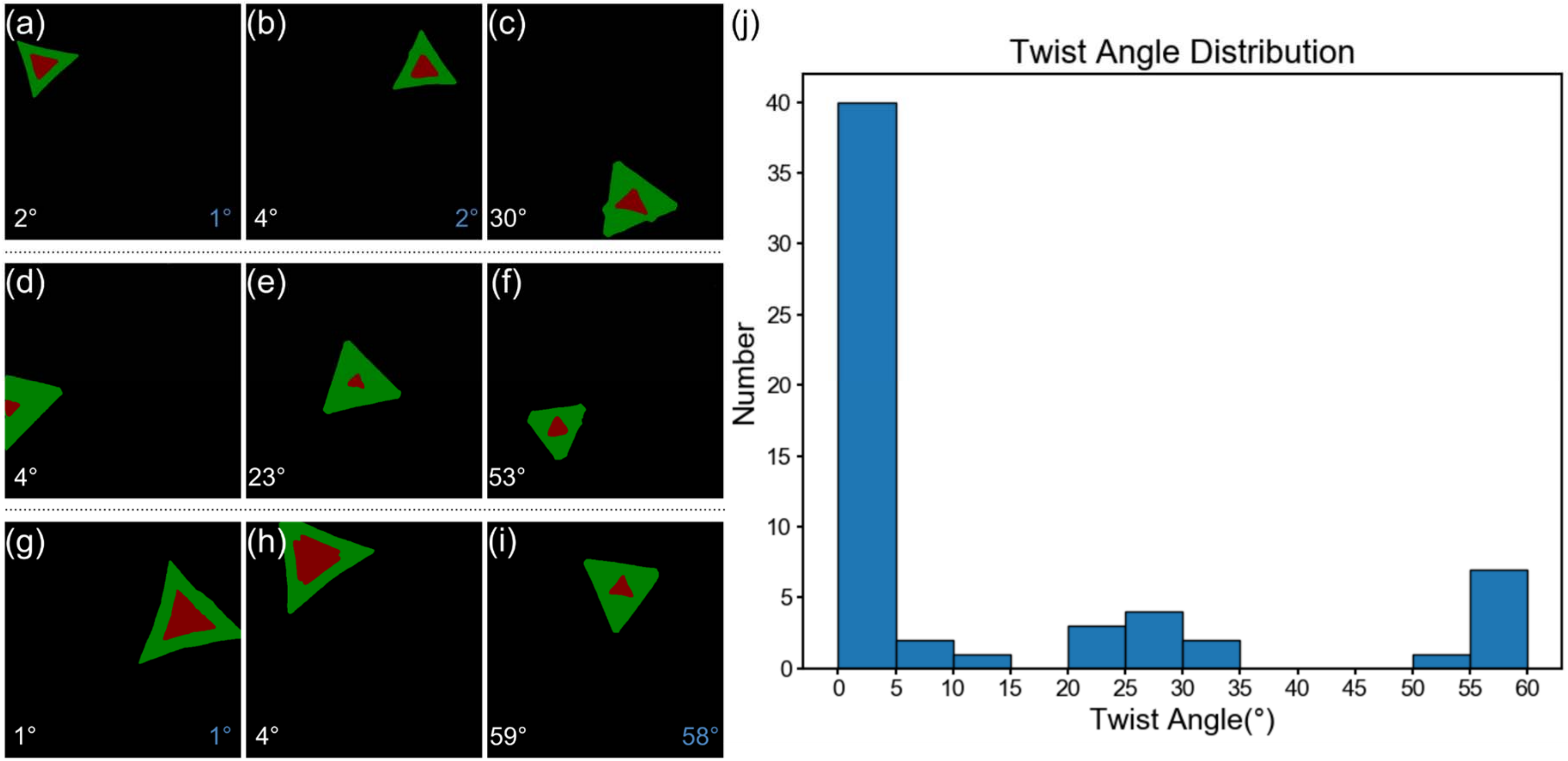
Deep learning approach for recognizing twist angles in MoS_2 flakes.

(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).

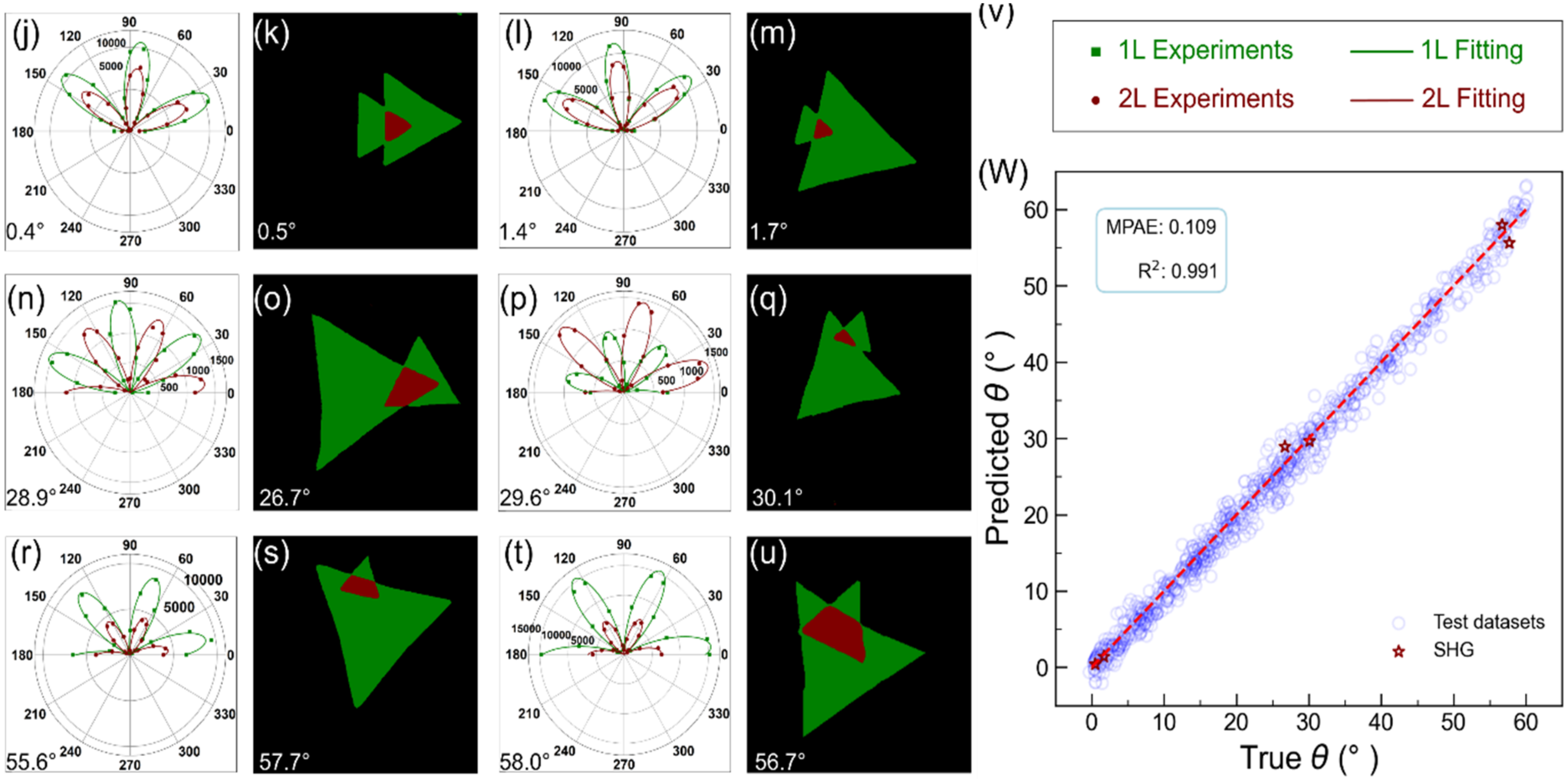
(c) Prediction of twist angles for actual as-grown MoS_2 bilayer samples post-segmentation

Deep Learning Predict Twist Angles



Performance evaluation of the twist angle Identification Model.

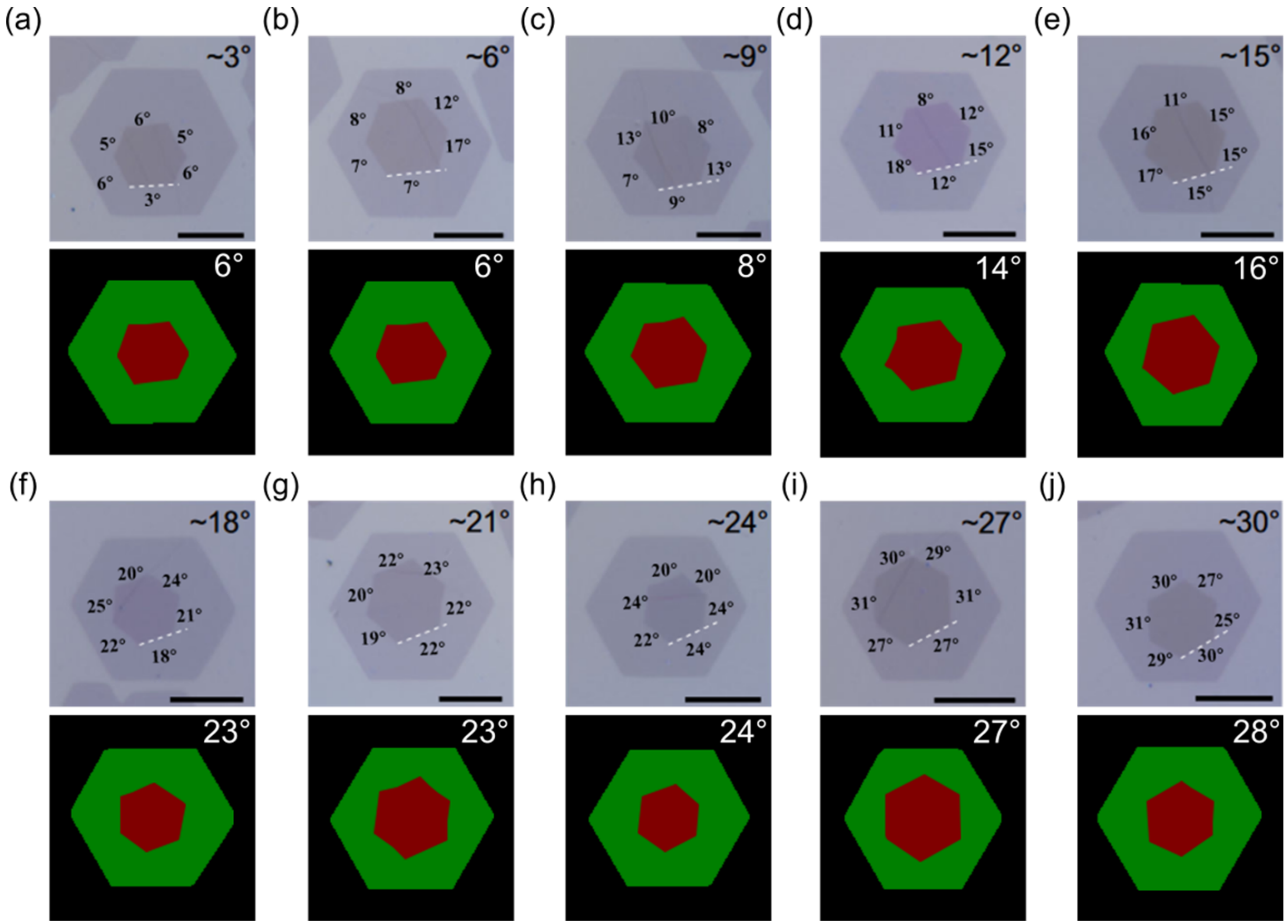
Deep Learning to Predict Twist Angles



Pingheng Tan

Nano Lett., 24, 9, 2789–2797 (2024)

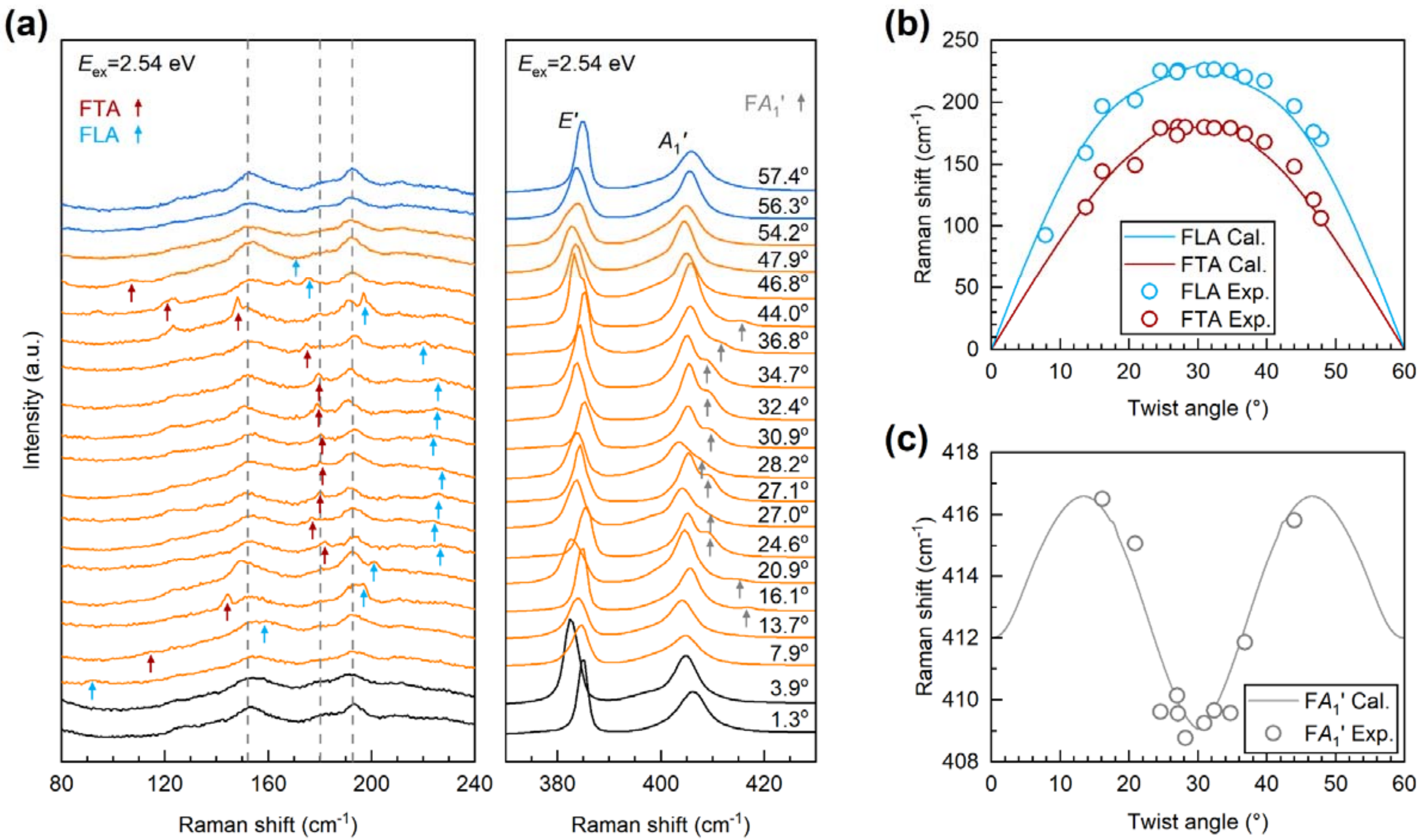
Deep Learning to Predict Twist Angles of Bilayer Graphene



Nat. Commun. 12, 2391 (2021)

Nano Lett., 24, 9, 2789–2797 (2024)

Moiré phonons in twisted CVD grown bilayer MoS_2



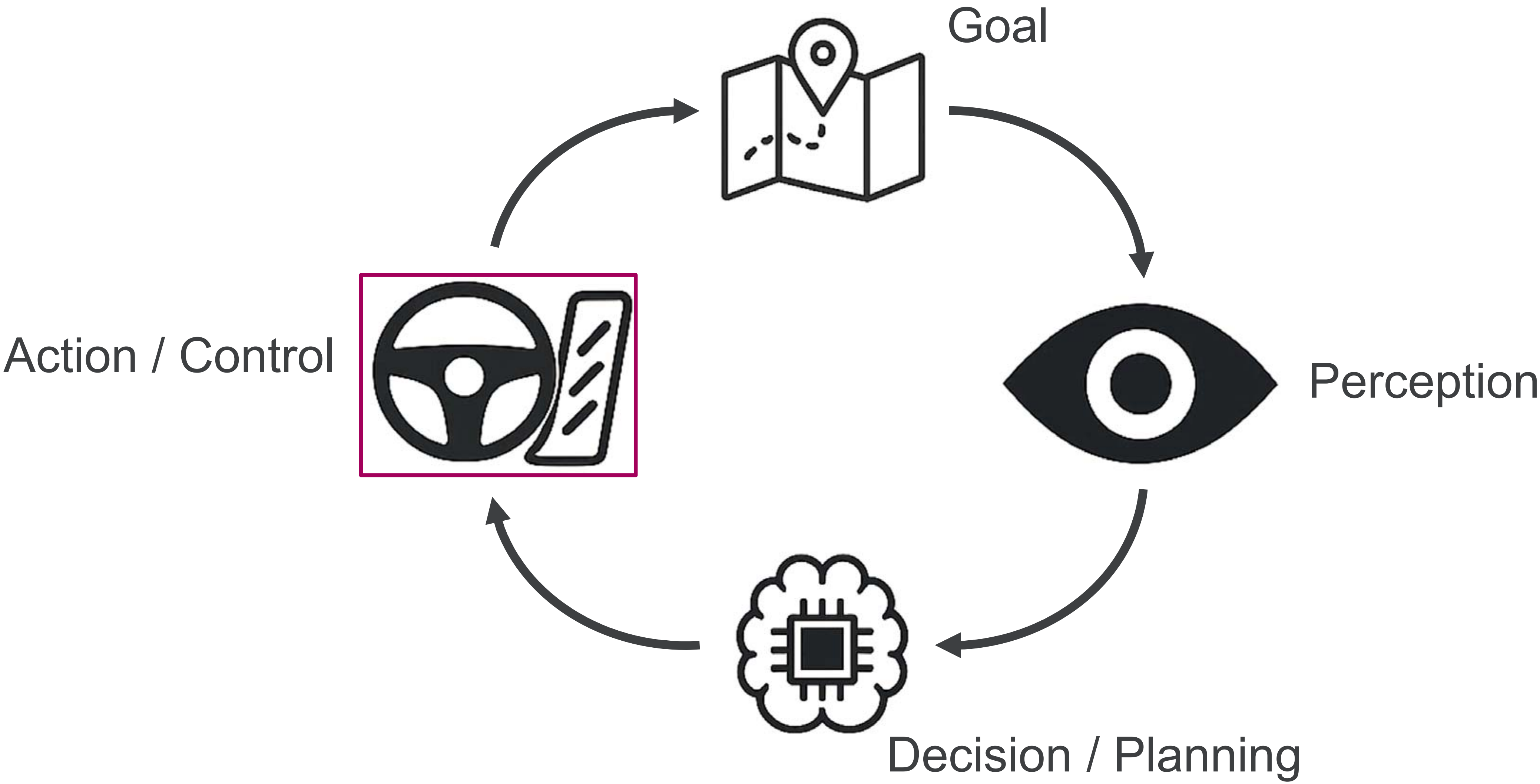
Pingheng Tan

Nano Lett., 24, 9, 2789–2797 (2024)

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

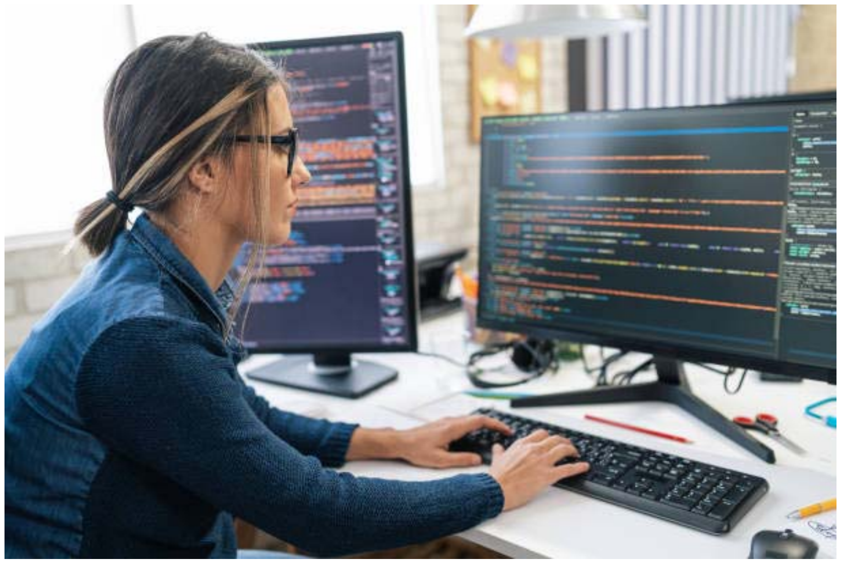
The concept of autonomous (self-driving) lab



Towards Autonomous Laboratory Instrumentation Control with LLM-based Tools

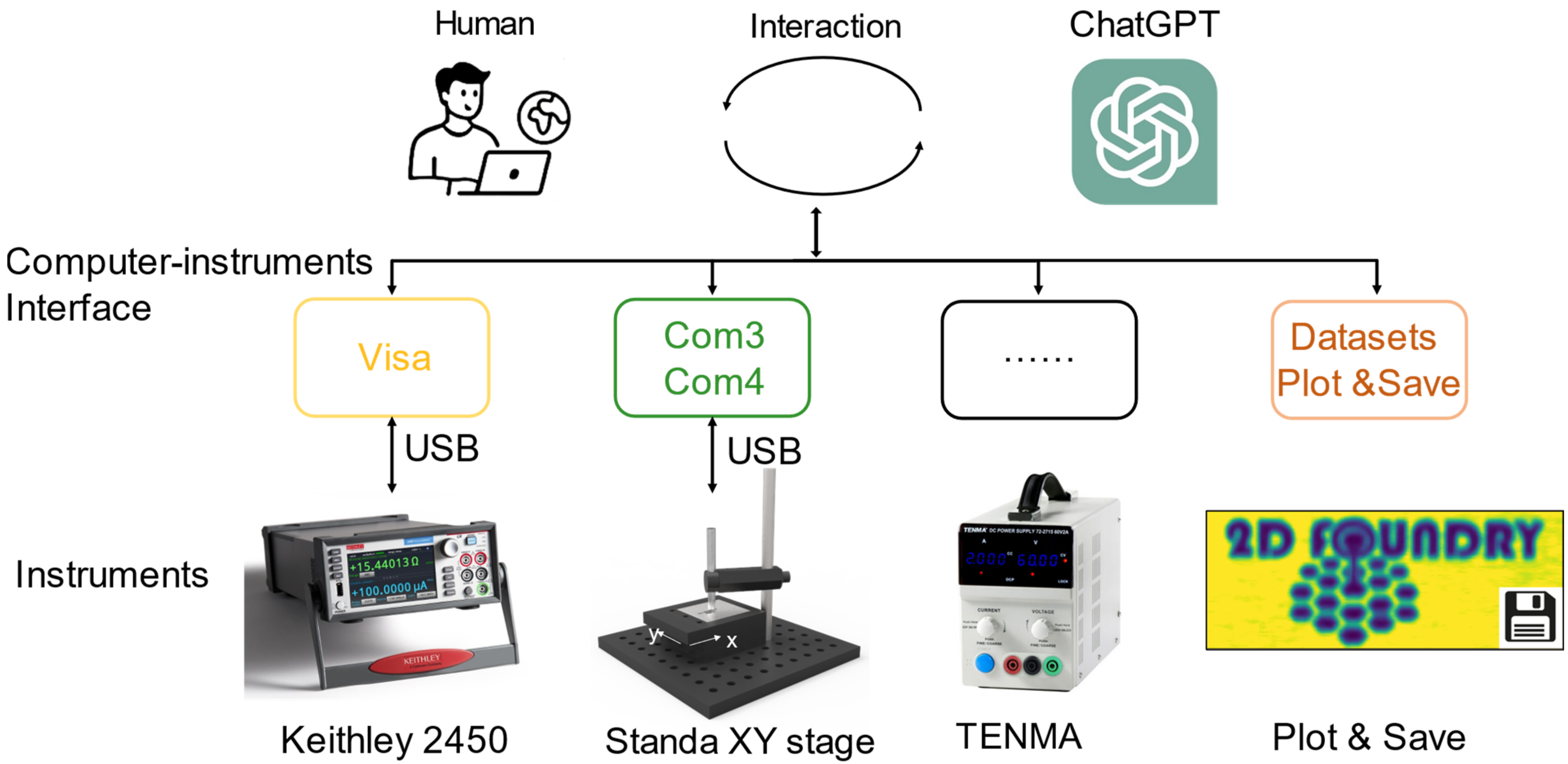
“Standard”

“Non – Standard”



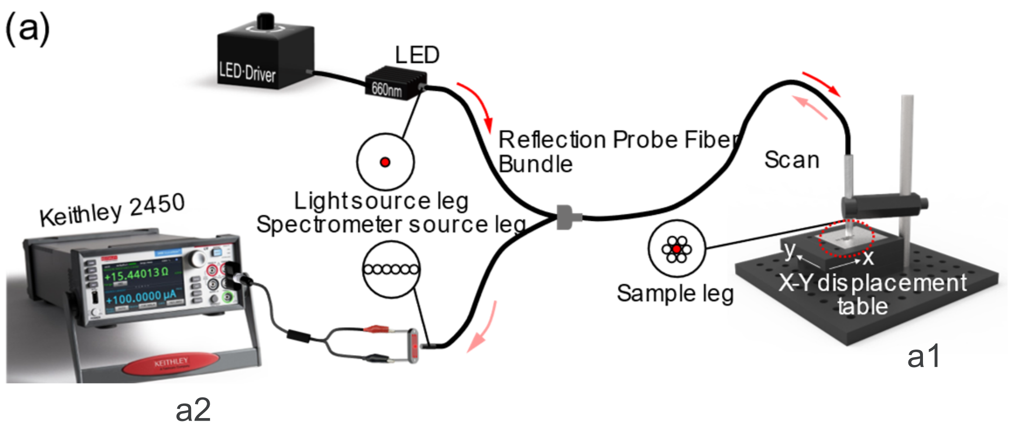
software developer

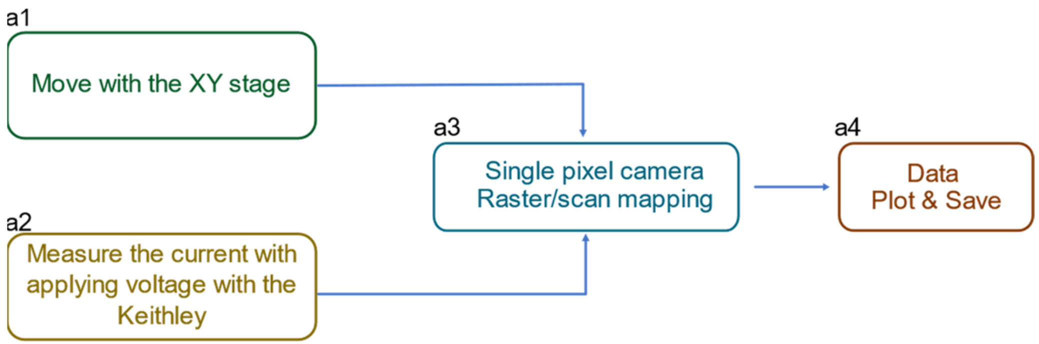
Towards autonomous laboratory instrumentation control with LLM-based tools



Small Struct. 6, 2500173 (2025)

Towards autonomous laboratory instrumentation control with LLM-based tools





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

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 afterwords. 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!

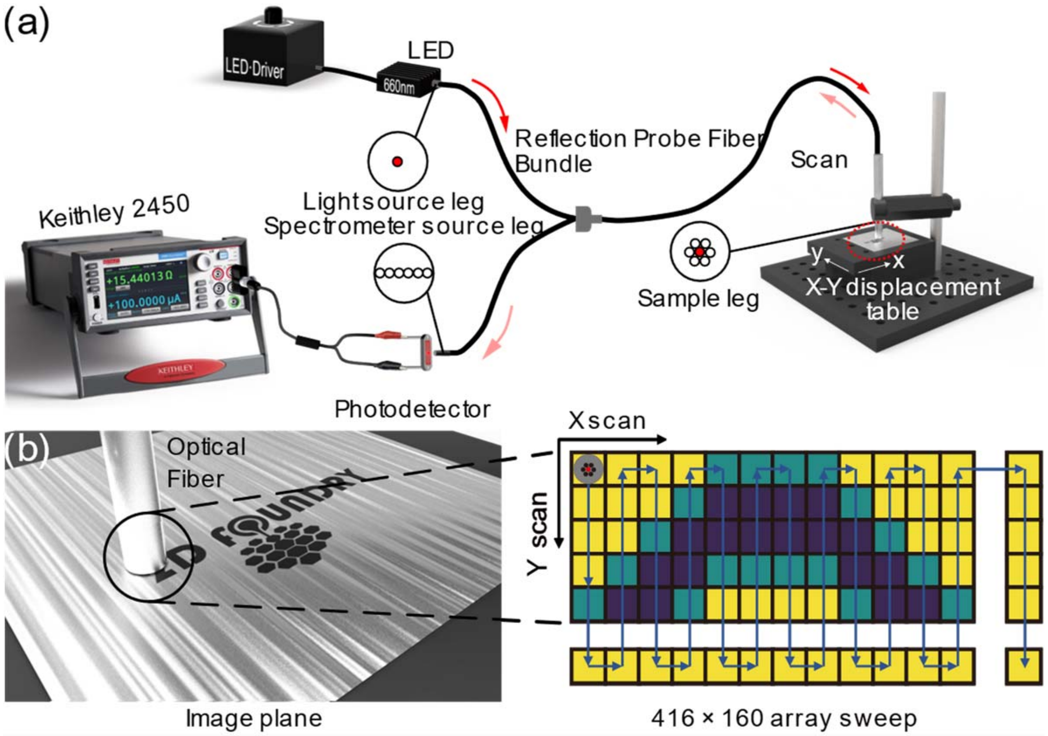
b4

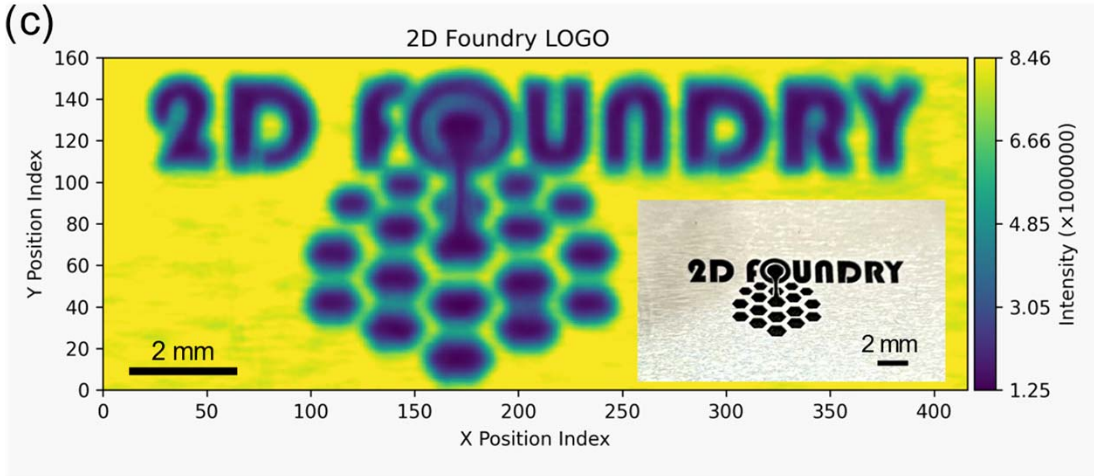
Prompt 4- input

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?**

Small Struct. 6, 2500173 (2025)

Towards autonomous laboratory instrumentation control with LLM-based tools





Single pixel camera

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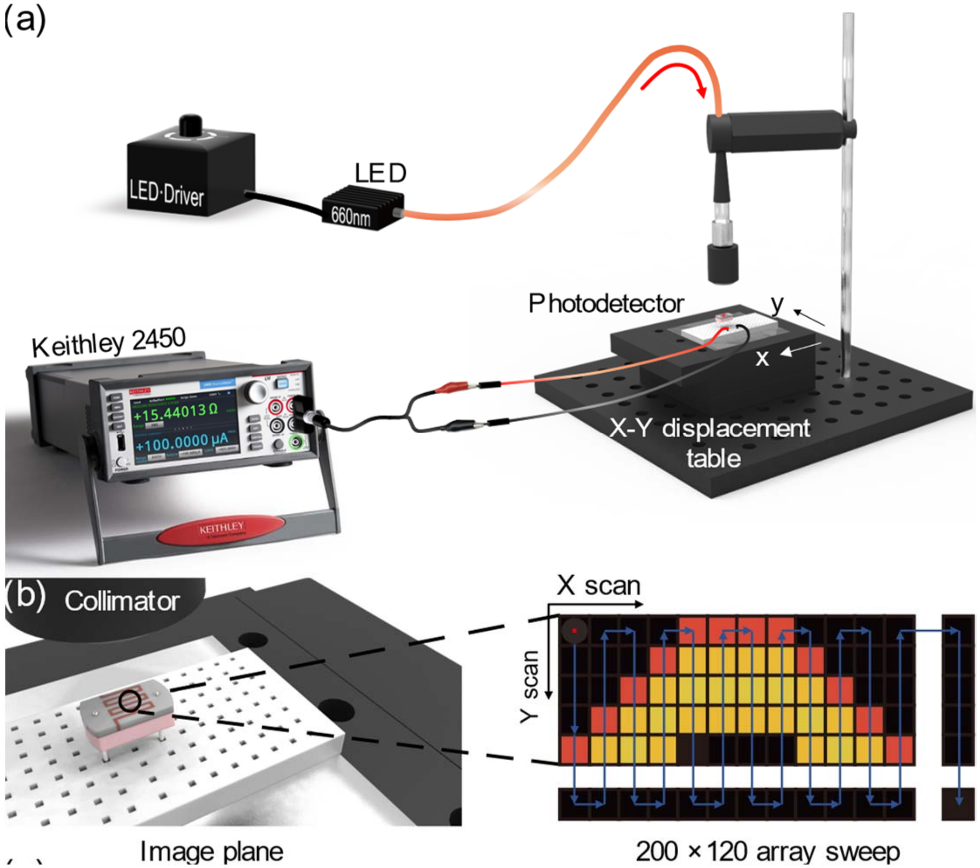
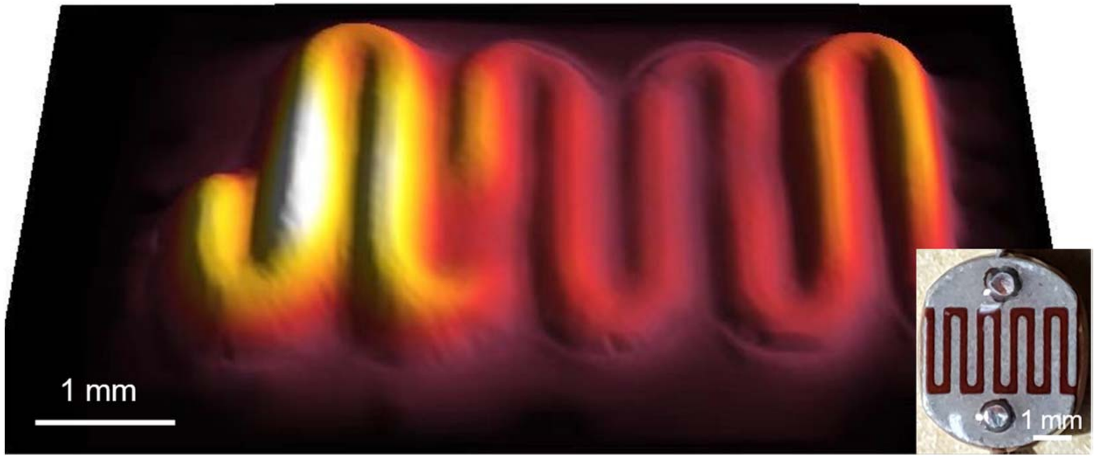
Towards autonomous laboratory instrumentation control with LLM-based tools



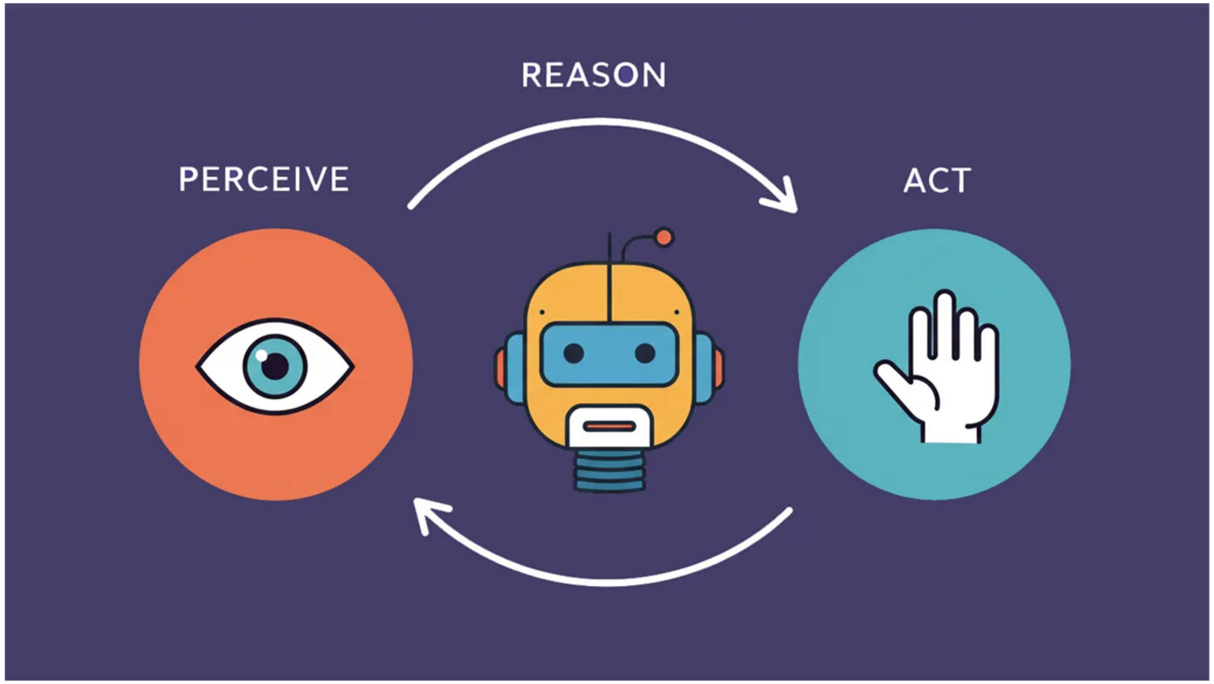


Scanning photocurrent mapping

Small Struct. 6, 2500173 (2025)

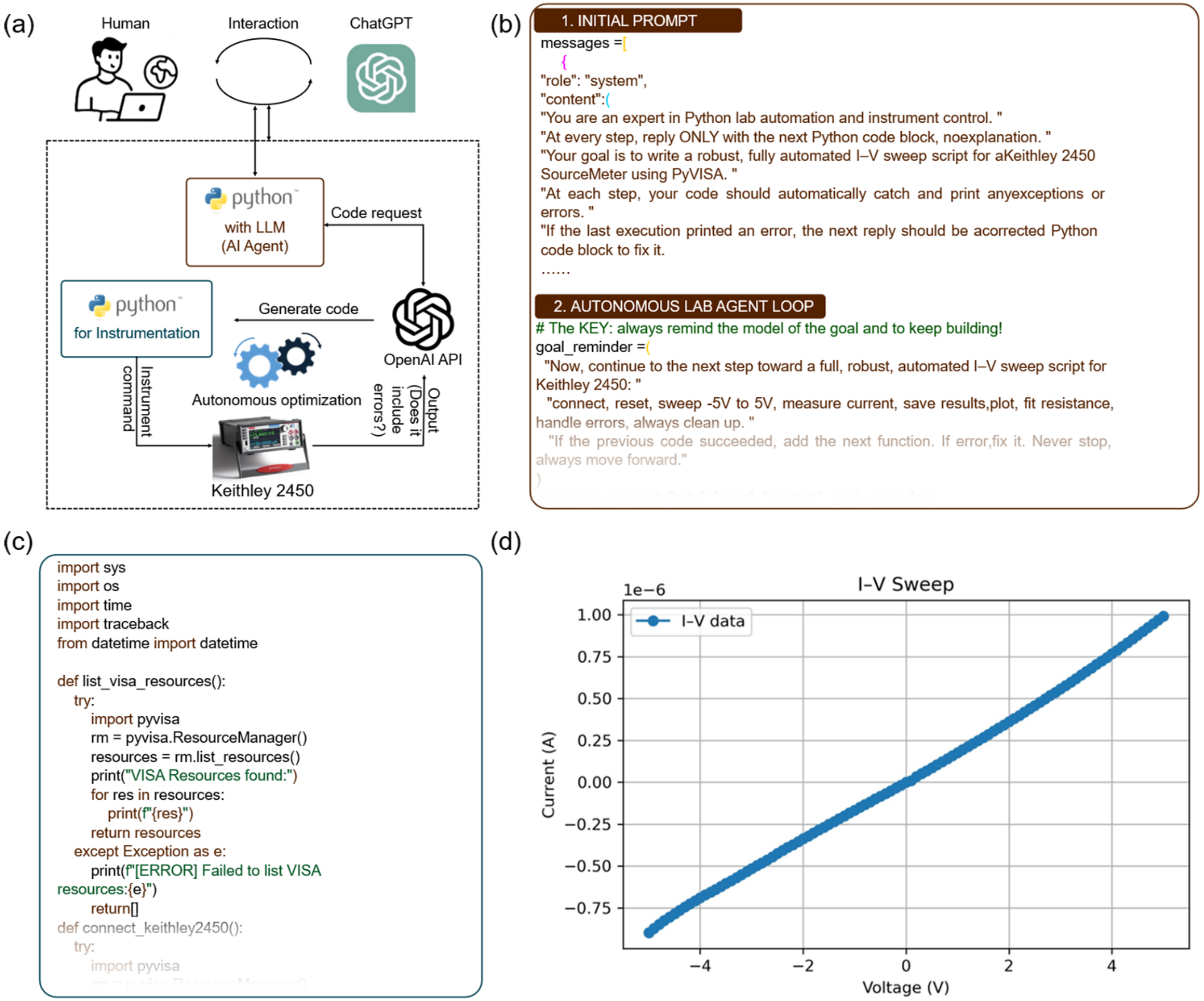
We were told that 2025 would be ***the year of agents***.

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

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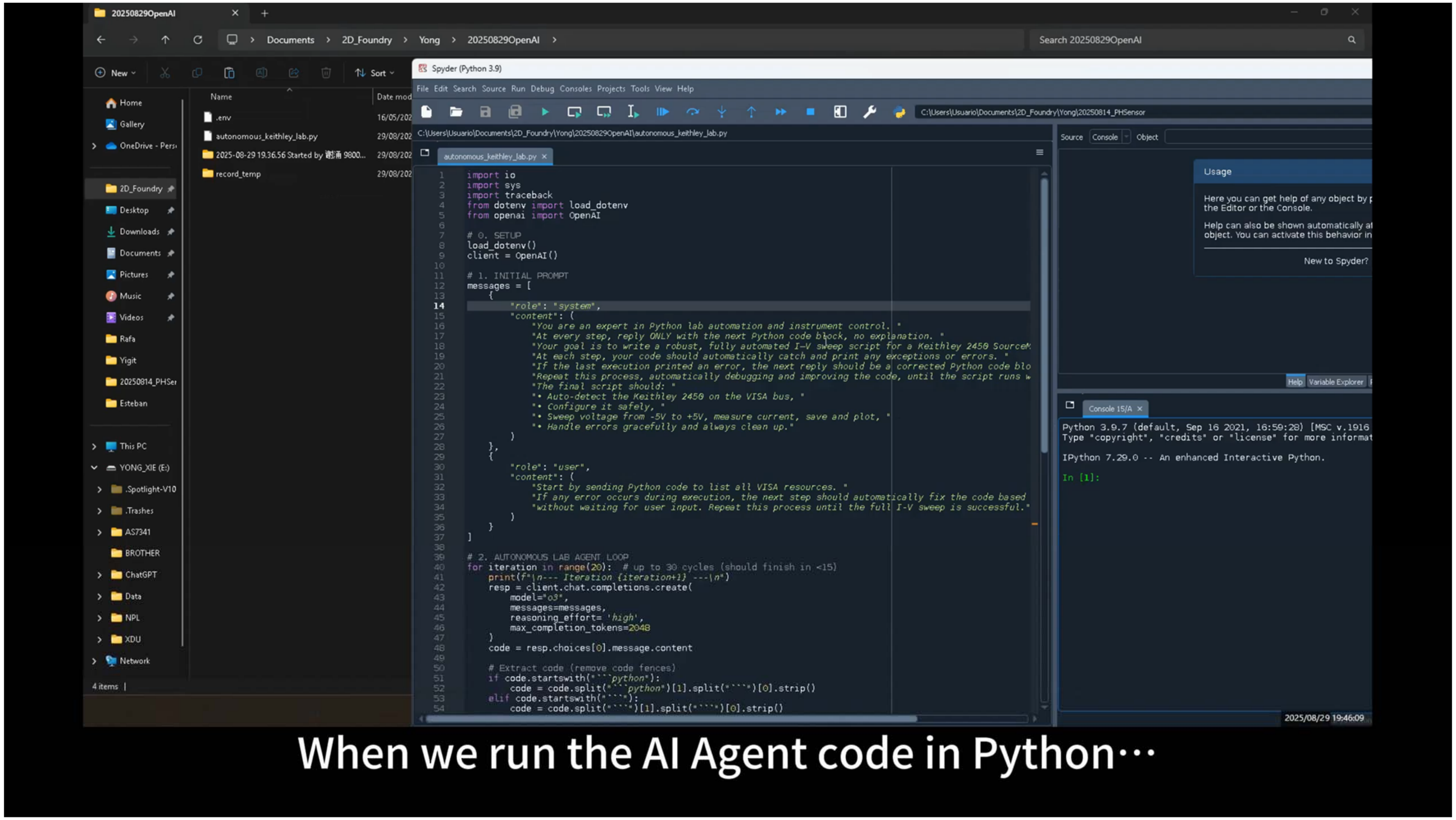
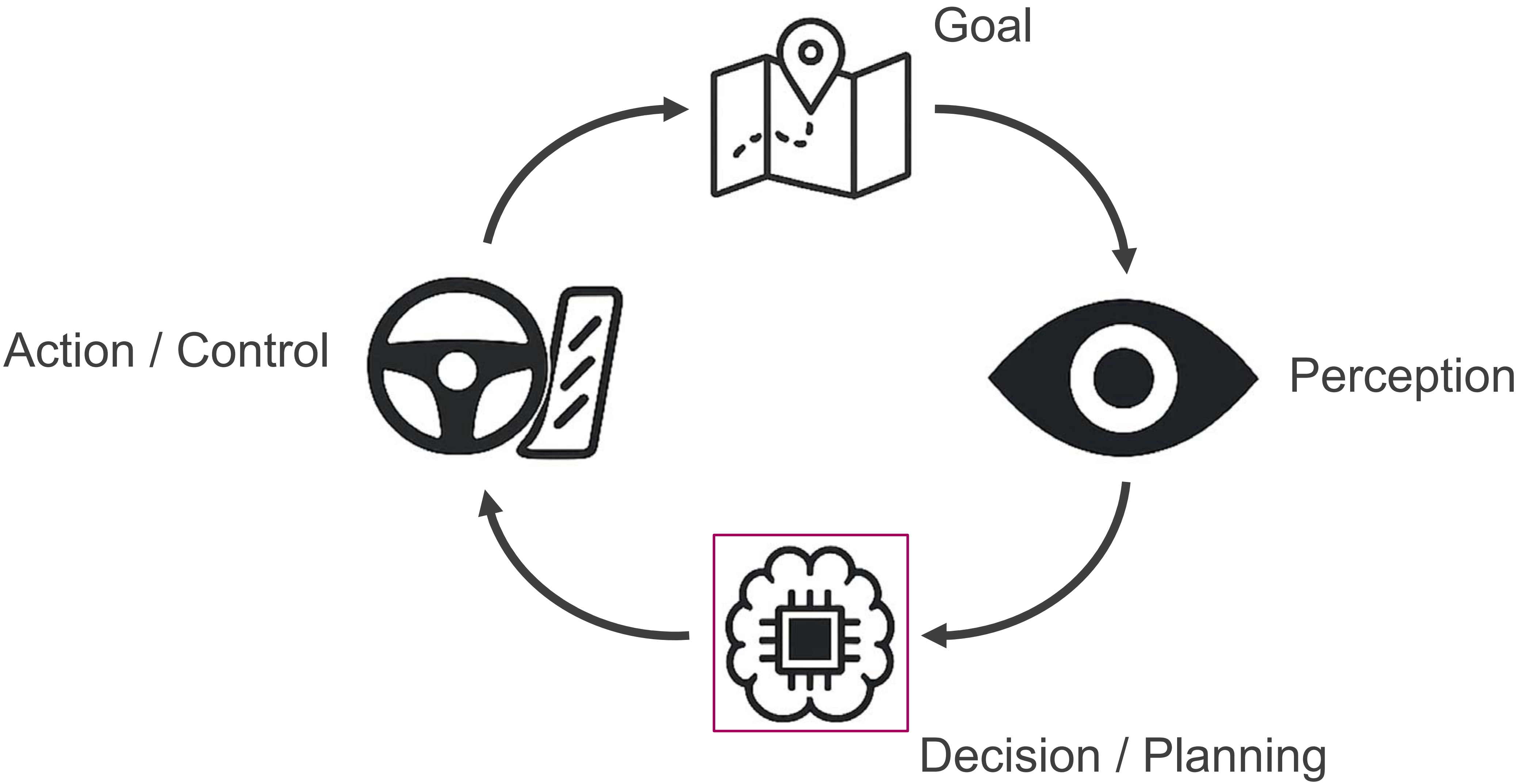
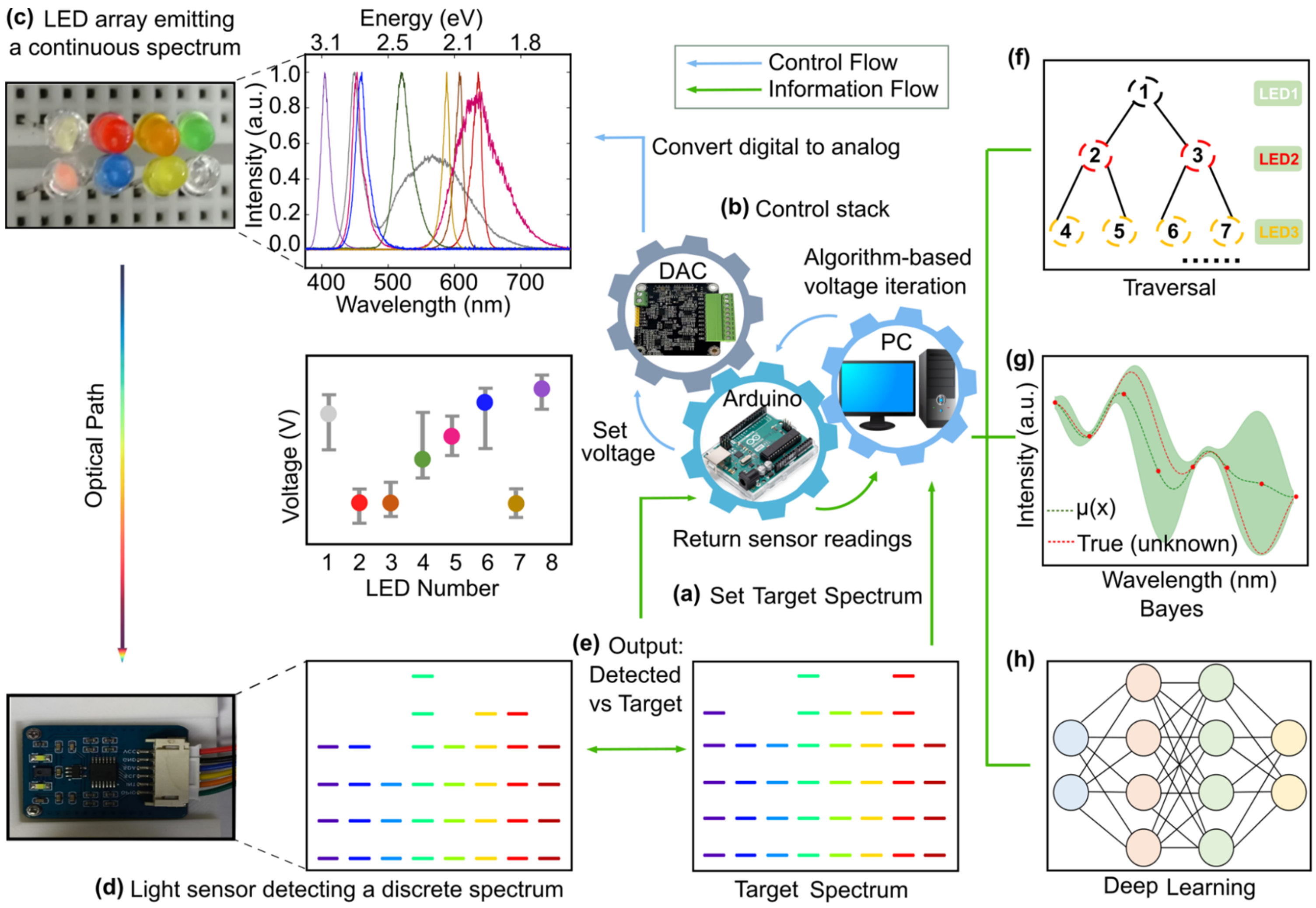

20250829OpenAI
Documents > 2D_Foundry > Yong > 20250829OpenAI
Spyder (Python 3.9)
autonomous_keithley_lab.py
Usage
Console 1/A
When we run the AI Agent code in Python…

The concept of autonomous (self-driving) lab

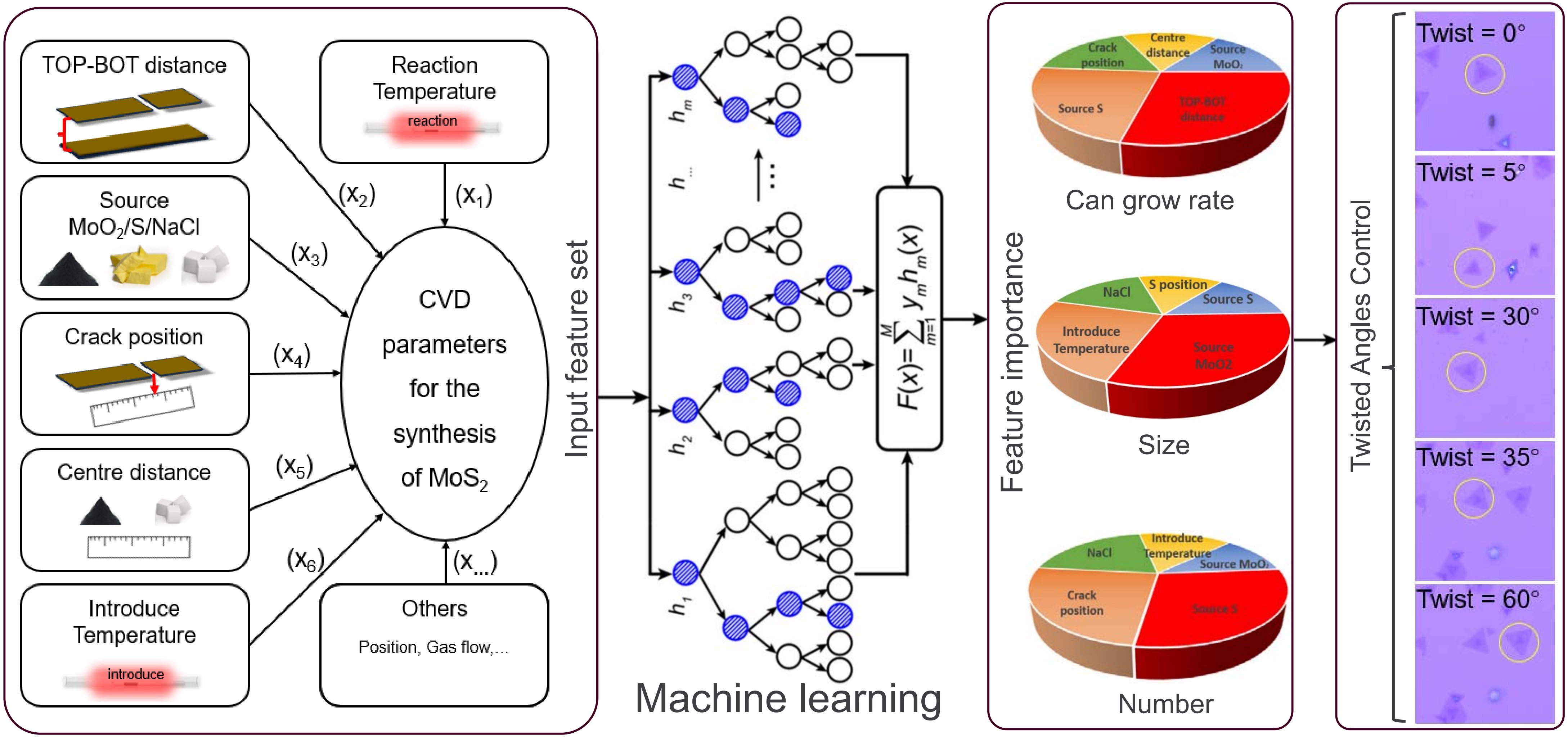


Towards autonomous laboratory



APL Machine Learning, revised

Towards autonomous synthesis for CVD 2D materials



In preparation

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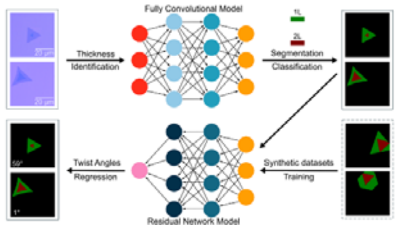
•ICMM: 2D Foundry group
•ICMM: Dr. Eduardo R. Hernández

•CAS: Prof. Pingheng Tan
Dr. Yan Zhou

•Students from Xidian University:
Q. Lei, H. Yang, X. Chen, K. He…

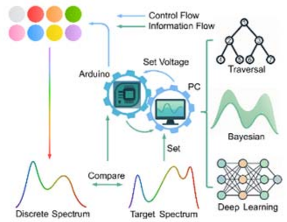
Take home message

- 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!