# 2D Materials Research in LMN

## Transfer of micromechanically exfoliated $MoS_2$ onto TEM-grids for defect characterization

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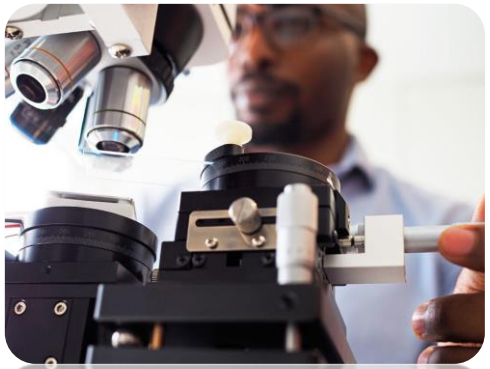


LMN SIEGEN

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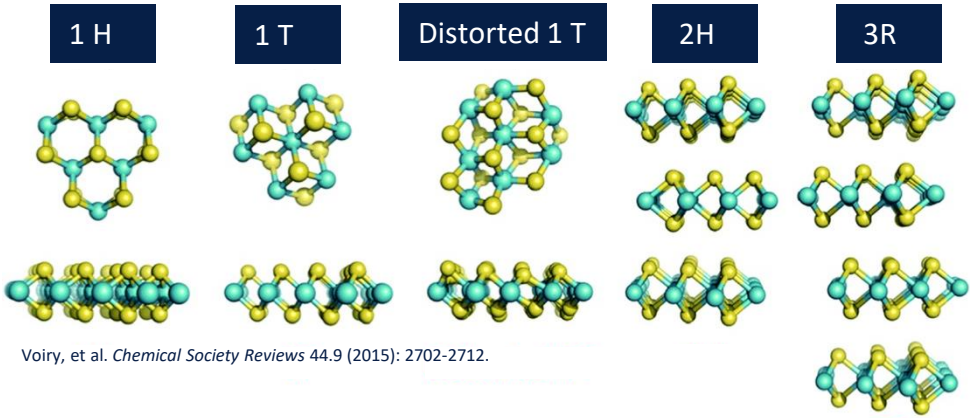
# Outline

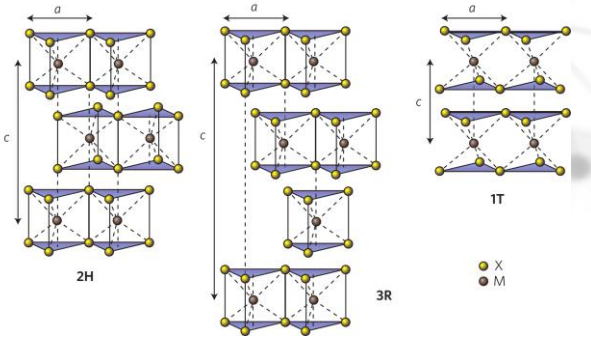
1. Introduction
    i. Defects in 2D transition metal dichalcogenides (TMDCs)
2. Defects characterization of (extended) defects
    i. Raman
    ii. TEM
3. Transfer to TEM – grids, the case of $MoS_2$
    i. Quantifoil® grids
    ii. Steel grids
    iii. $Si_3N_4$

# 2D materials: (X-M-X) TMDs

$MX_2$ ( M = Mo, W; X = S, Se) structural polytypes




Voiry, et al. *Chemical Society Reviews* 44.9 (2015): 2702-2712.

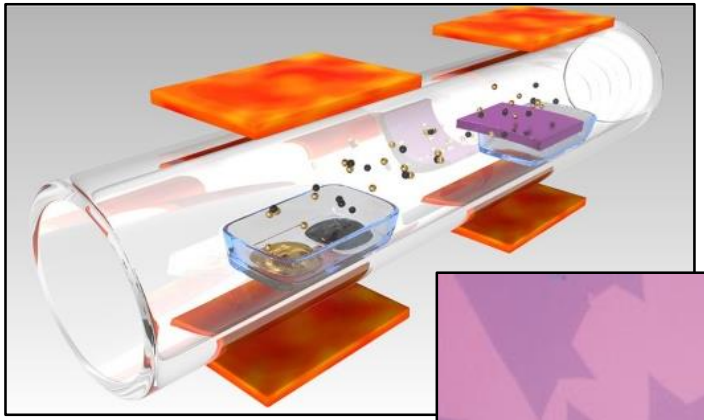



Wang, et al. *Nature nanotechnology*, 2012, 7. Jg., Nr. 11, S. 699-712.

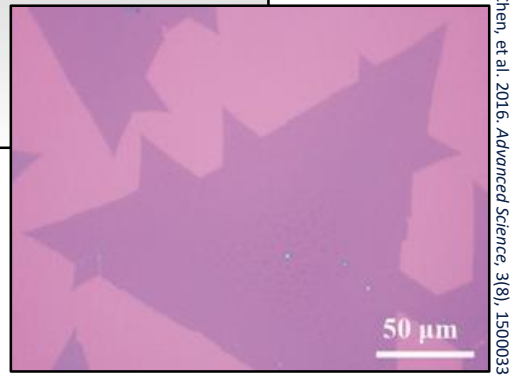
→ Confers the material important properties: metallic, semiconducting character, and stability

# $MoS_2$ common synthesis routes

## Bottom-up



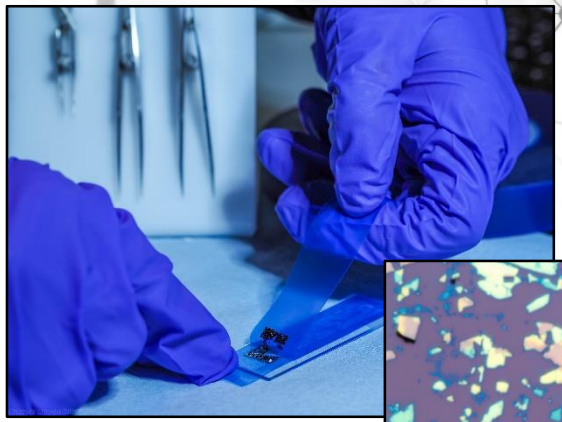
Jung, et al.2015. *Scientific Reports*, 5(1), 1-9.

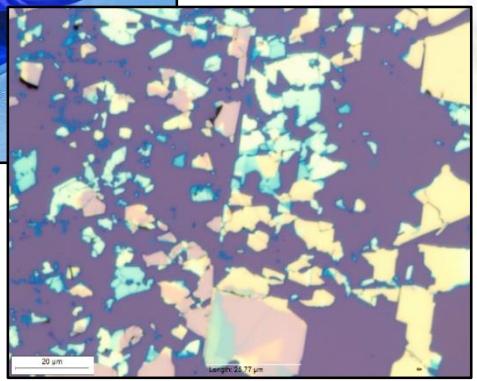



Chen, et al. 2016. *Advanced Science*, 3(8), 1500033.

- Layer-by-layer growth
- Large scale production
- Limited quality

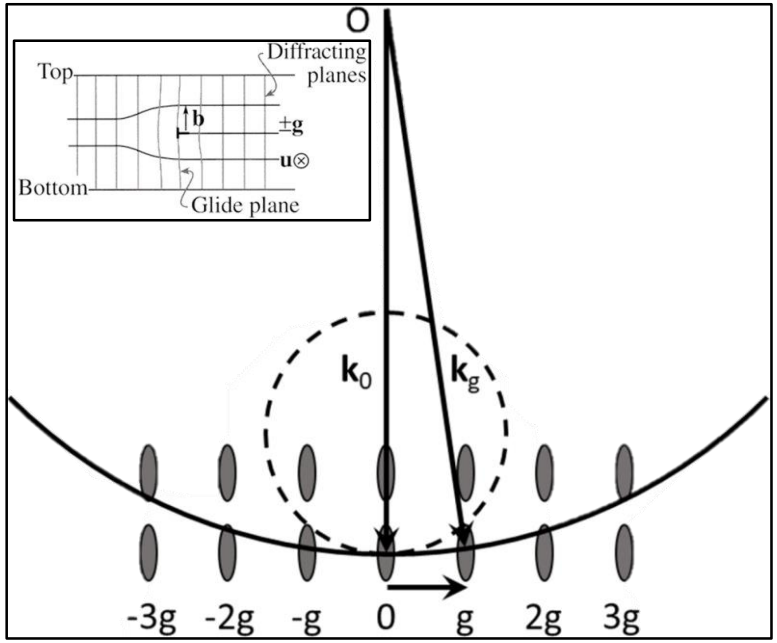
## Top-down





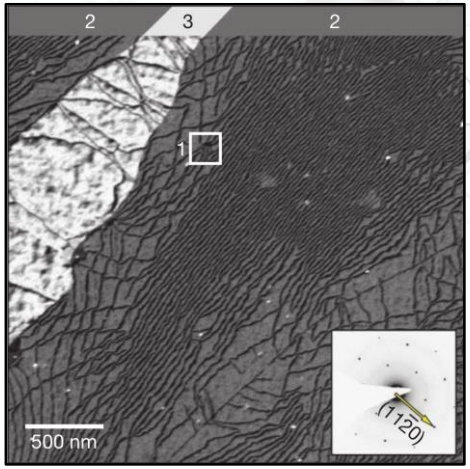
- High quality but low yield
- Large area flakes
- Pristine properties

# Dislocations visualization using DF-TEM




Williams & Carter. Springer, 2009.

## Dislocations in B(T)LG




Butz, et al. *Nature.* 505.7484 (2014): 533-537.

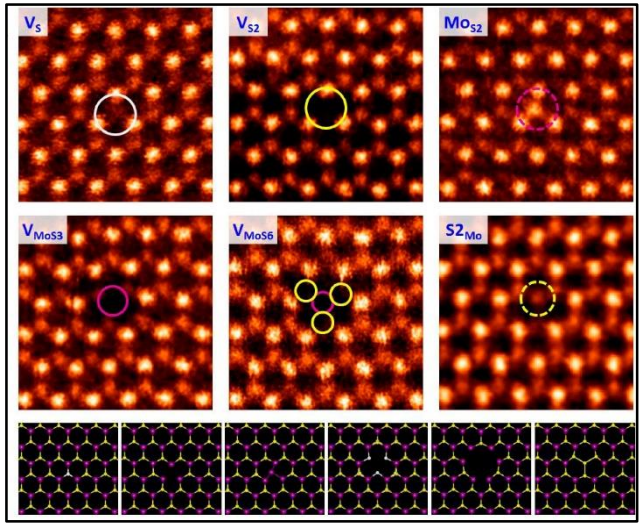
Invisibility criterion

$\vec{g} \cdot \vec{b} = 0$ → screw dislocations

$\vec{g} \cdot \vec{b} \times \vec{u} = 0$ → edge dislocations

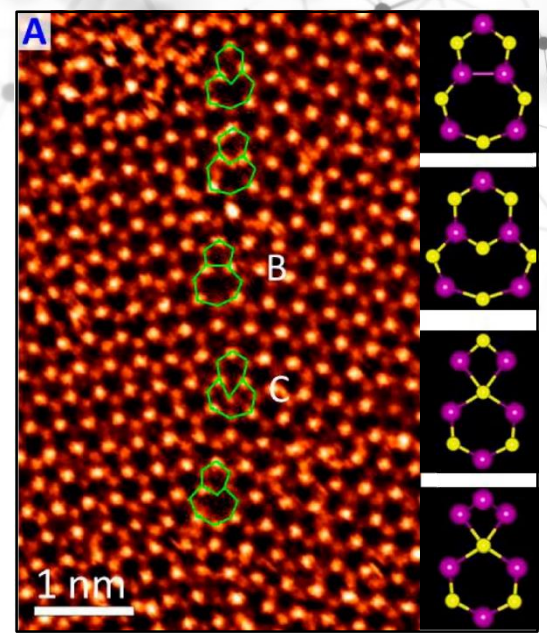
# STEM-ADF for defects characterization in monolayer CVD $MoS_2$

In Literature:




Zhou, et al. Nano Letters 13, 2615-2622, doi:10.1021/nl4007479 (2013).

Point defects in CVD ML $MoS_2$




GBs in $MoS_2$

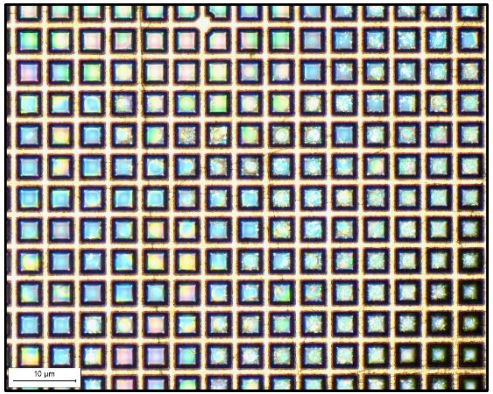
For extended defects:

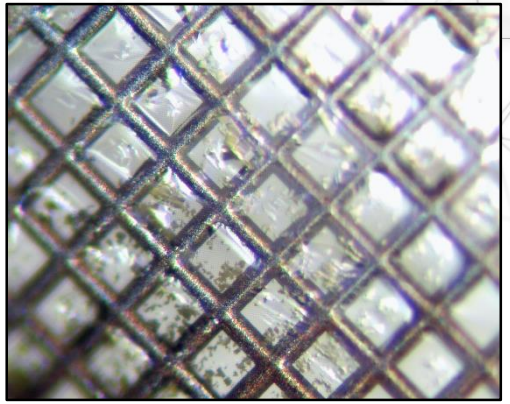
- Dislocations, solitons, corrugations, Moiré lattices, stacking domain walls, ripplocations??

☛ Lack of comprehensive systematic characterization of extended defects in TMDCs

# From exfoliation to TEM-grid

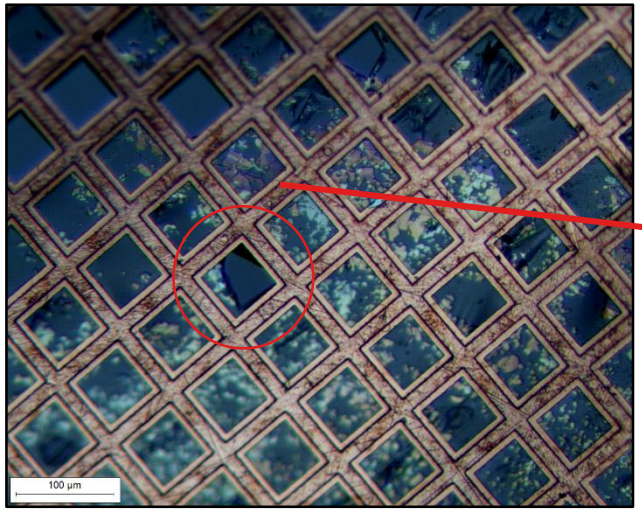
## Transfer to Quantifoil® R1.2/1.3 grids

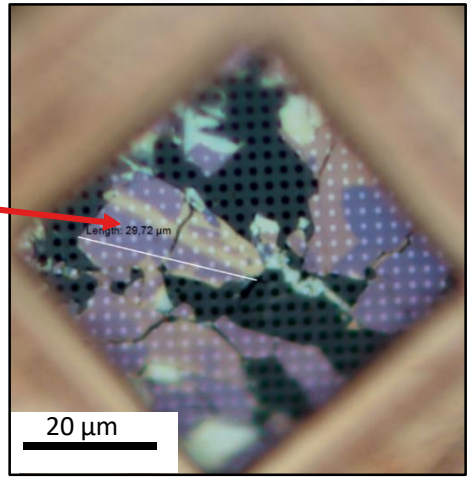


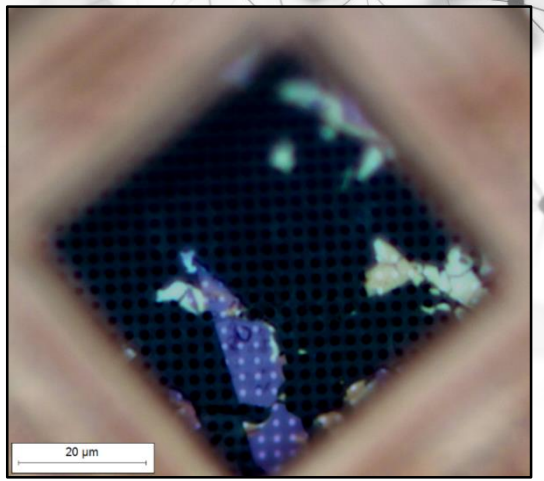


- Exfoliate
- Transfer onto 285 nm $SiO_2$/Si wafer
- Attach TEM grid with IPA
- Spincoat 5 wt% PC
- Peel off
- Thoroughly wash with chloroform

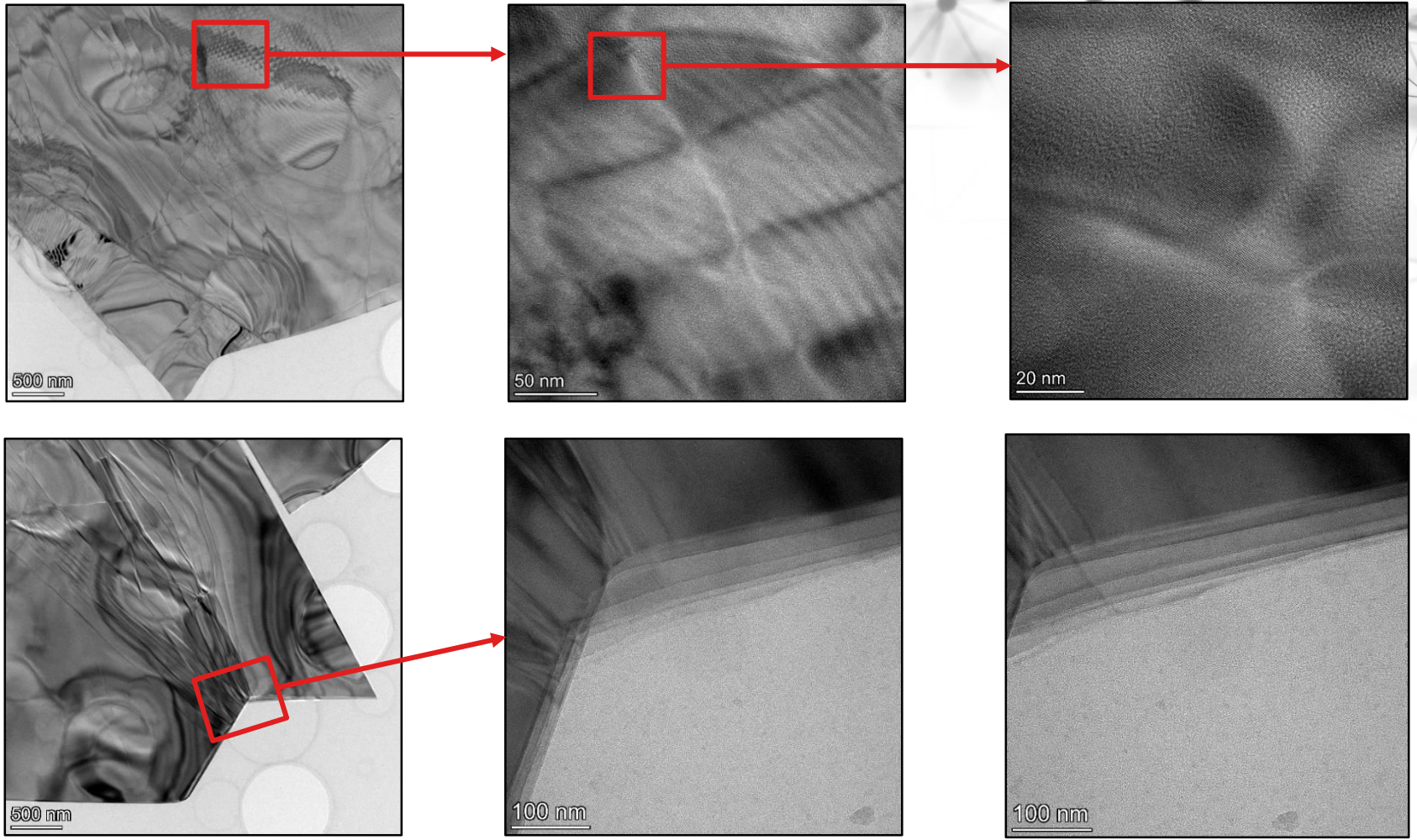
# Quantifoil ® R1.2-1.3 – Challenges



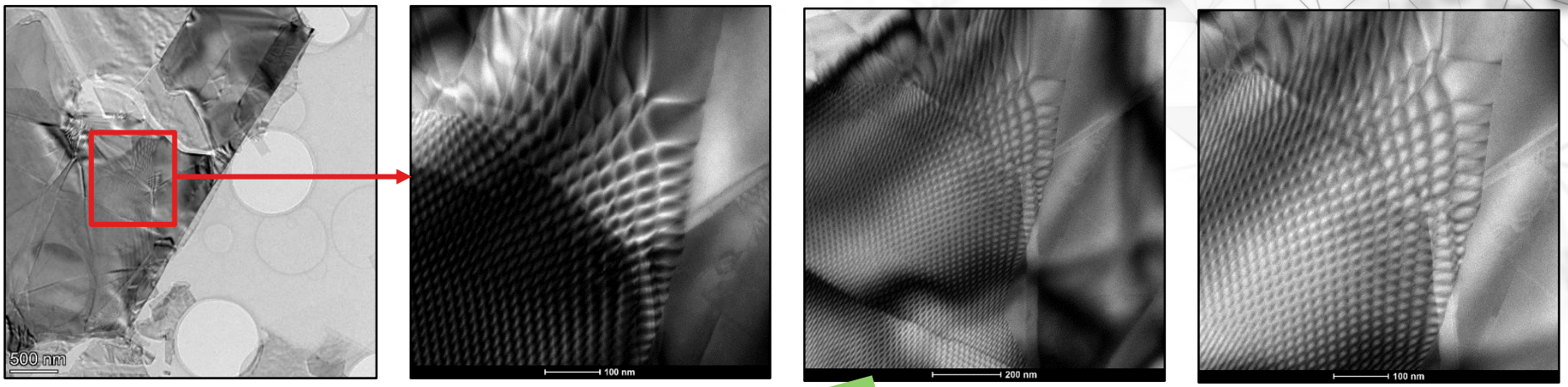




- Rupturing of the support membranes(high surface tension due to rapid uncontrolled solvent evaporation.
- Controlled by slow drying between solvent processes (critical point dryer)
- 5-10 % rupturing even when slow-dried


500 nm
50 nm
20 nm
500 nm
100 nm
100 nm

# DF TEM contrast variation from different reflexes



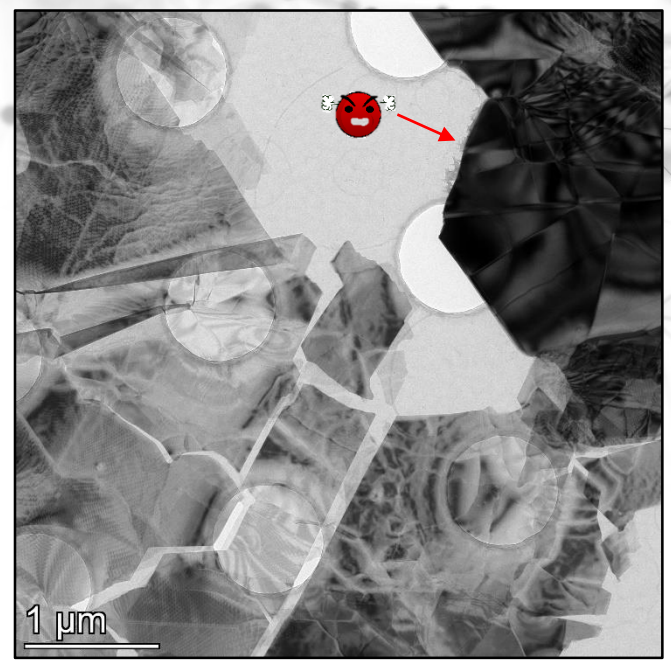

- Regular dense dislocation networks
- Rotational Moiré networks

Coming up…

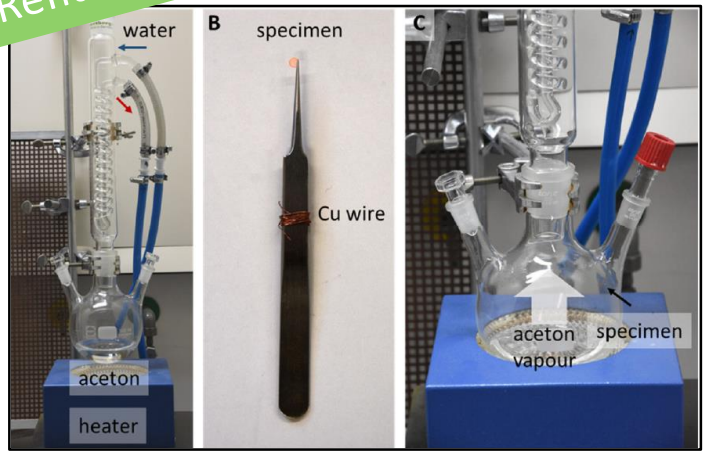
- Corresponding diffraction patterns
- Exciting reflexes under the 2-beam condition (non-trivial)
- Detailed dislocations characterization in few layer/bilayer $MoS_2$

# Dealing with the contamination!!

- Hydrocarbon contamination
  - PC
  - IPA
- Quantifoil adsorbed contaminants from ambient air
- Polymer residues: Tape, PC

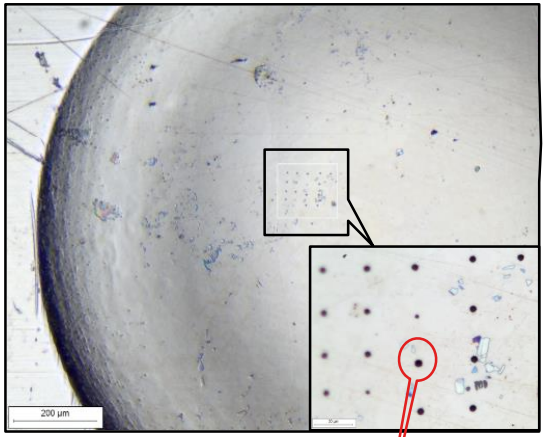


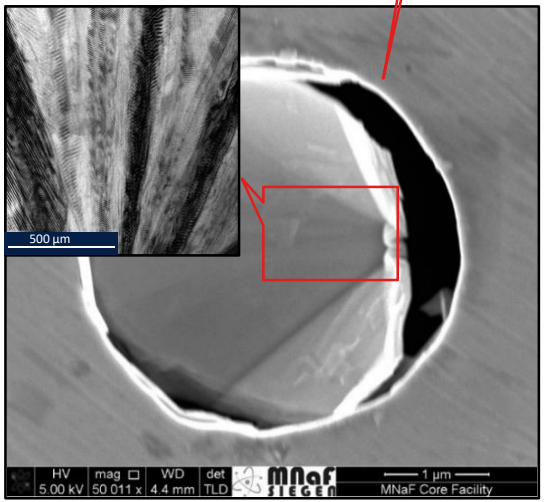

Refluxing

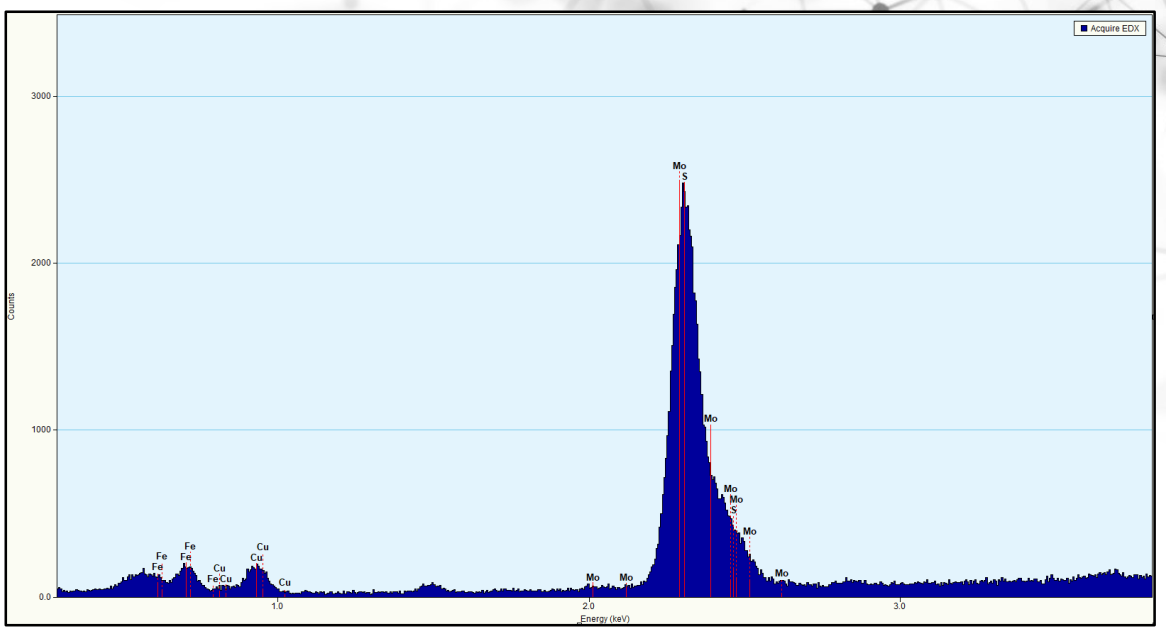



CPD

# Transfer to TEM grids option 2: Transfer to stable grids



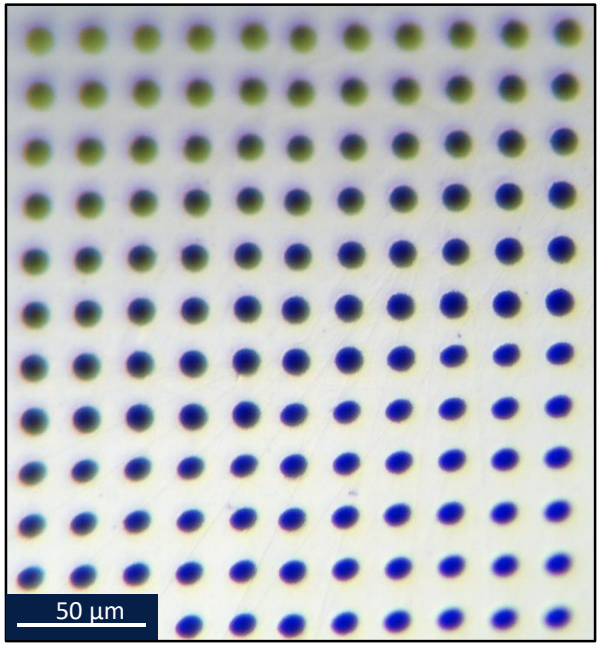




- Direct exfoliation first try
- Challenge in milling (here 50 µm thick)
  - Further grinding
  - Dimpling

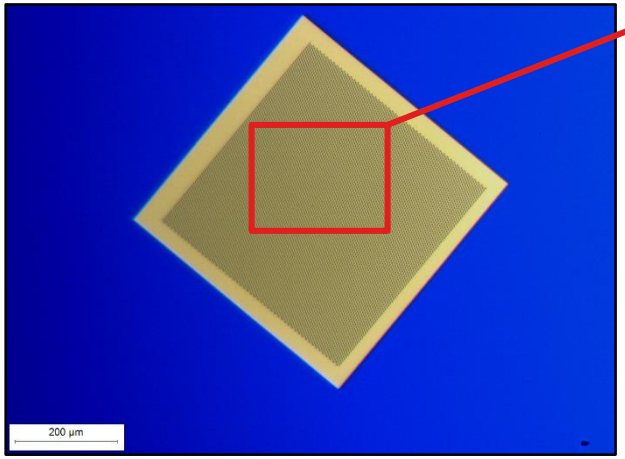
→Promising initial findings w.r.t sample cleanliness

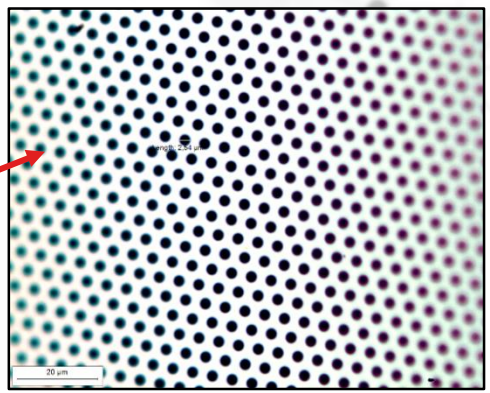
What's new!!!

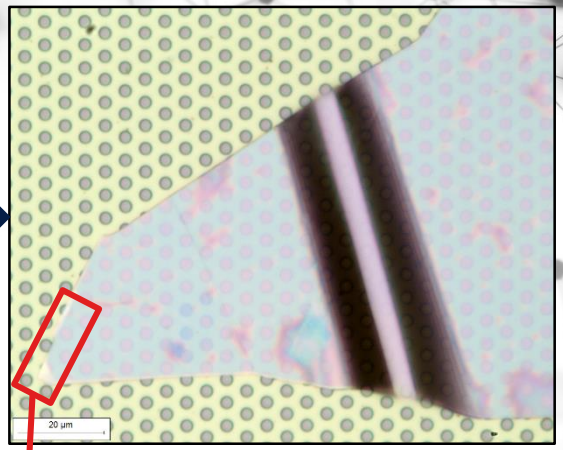


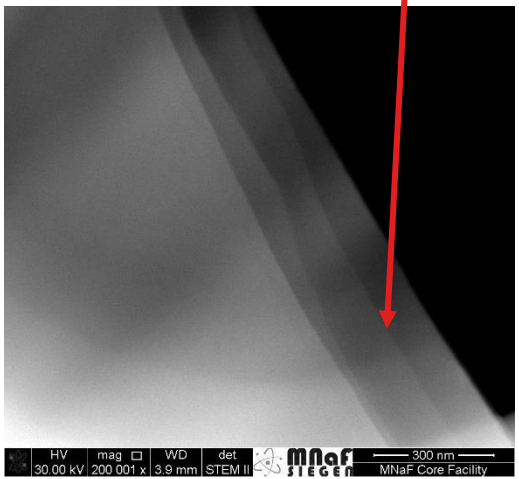

- 8 µm
- 16 (± 2) µm stainless steel grid with a 3 mm circular frame
- Ø of the holes is 8 µm, pitch 10 µm
- 12 x 12 raster
- Solvent resistant
- Can be plasma-cleaned
- Background is C-free
- 7 minutes per hole – milling time
- Backing at higher temperatures possible

# Transfer to TEM grids : Stamping on $Si_3N_4$ membranes




- Solvent-free transfer
- PDMS-based dry transfer

→ Minimize introduction of impurities

THANK YOU FOR YOUR ATTENTION !!!