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# Synthesis and characterization of CoCrNi and $\text{Fe}_x(\text{CoCrNi})_{1-x}$ high entropy alloy thin films with unique atomic structures and enhanced mechanical properties

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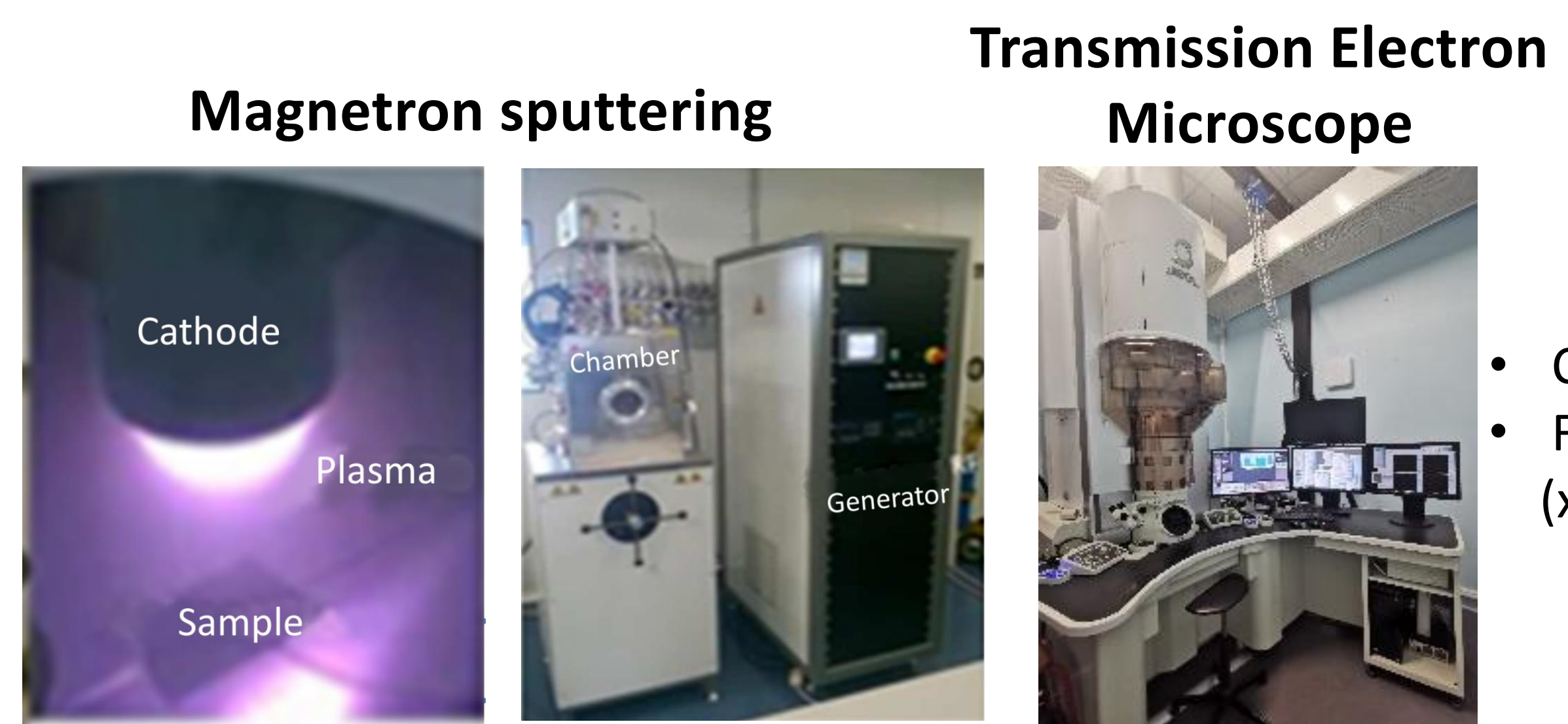
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## Context & Objective

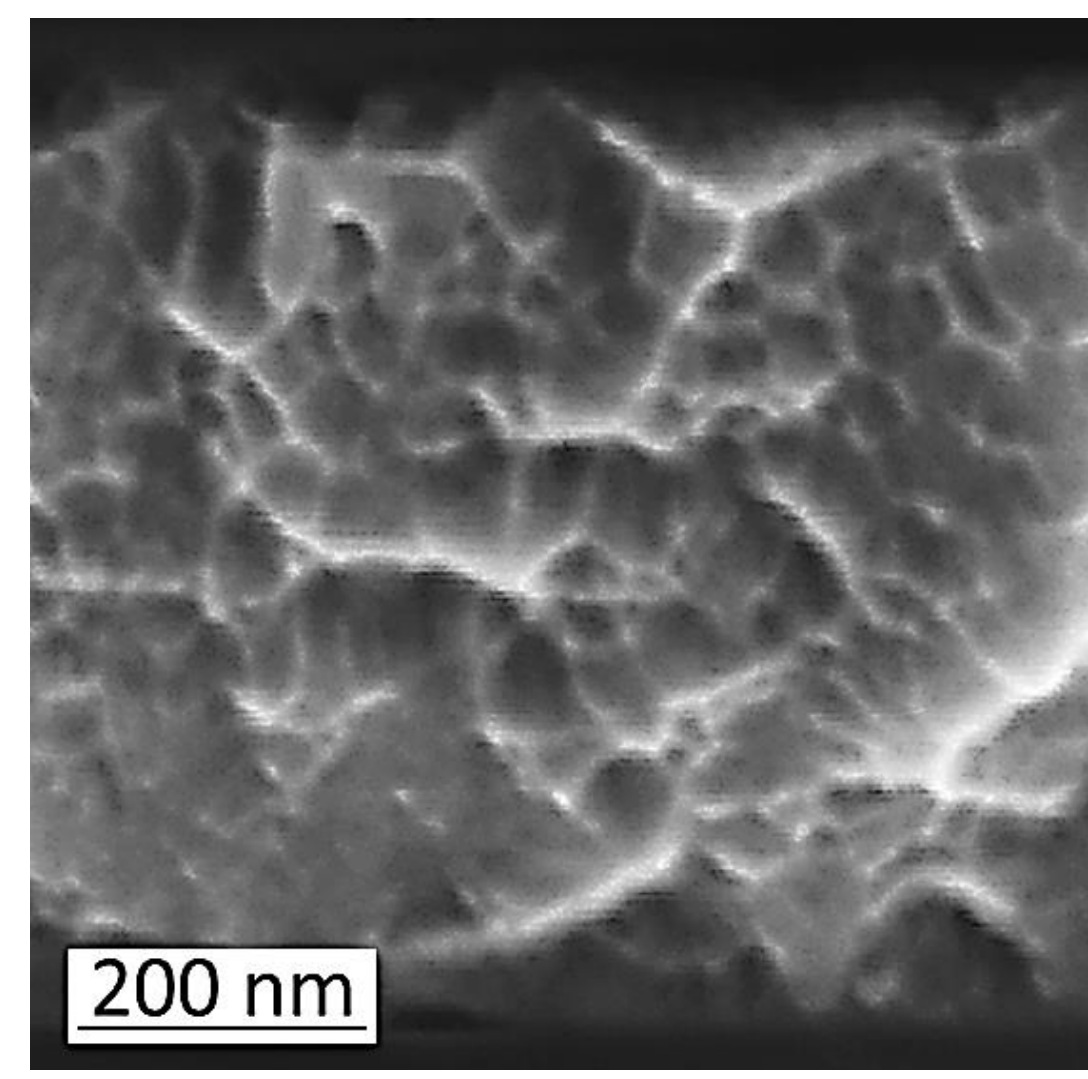
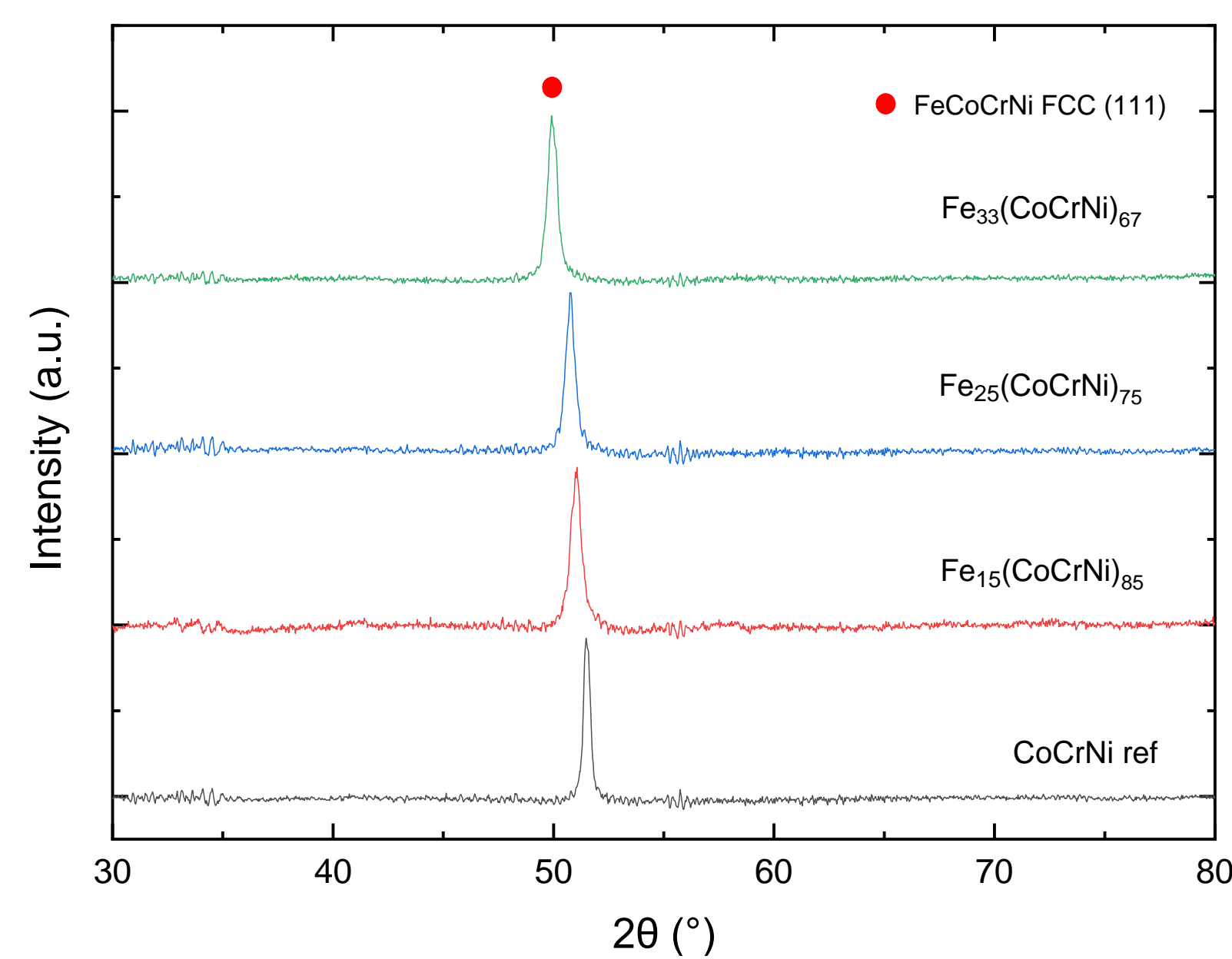
Compositionally complex CoCrNi thin films have reported enhanced values of hardness (up to 10 GPa) and elastic modulus (up to 250 GPa), due to a peculiar nanotwinned structure and columnar grains with coherent twin boundaries [1]. The addition of Fe – to form  $\text{Fe}_x(\text{CoCrNi})_{1-x}$  – leads to a further enhancement of the mechanical properties (Hardness >11 GPa) due to the stabilization of the FCC phase and the formation of a Cr-rich  $\sigma$  phase [2] with a small grain size of ~20 nm [3]. However, studies on the film growth mechanisms and the relationship of atomic-microstructure and mechanical behaviour have been poorly explored. Here, we deposited CoCrNi and  $\text{Fe}_x(\text{CoCrNi})_{1-x}$  thin films with controlled thicknesses from 15 up to 700 nm, to investigate the effect Fe on the atomic structural evolution, the relationship with mechanical properties, while exploring early-stage film growth regime.



- ### Compositions
- CoCrNi
  - $\text{Fe}_x(\text{CoCrNi})_{1-x}$  (x= 0, 15, 25, 33 at.%)

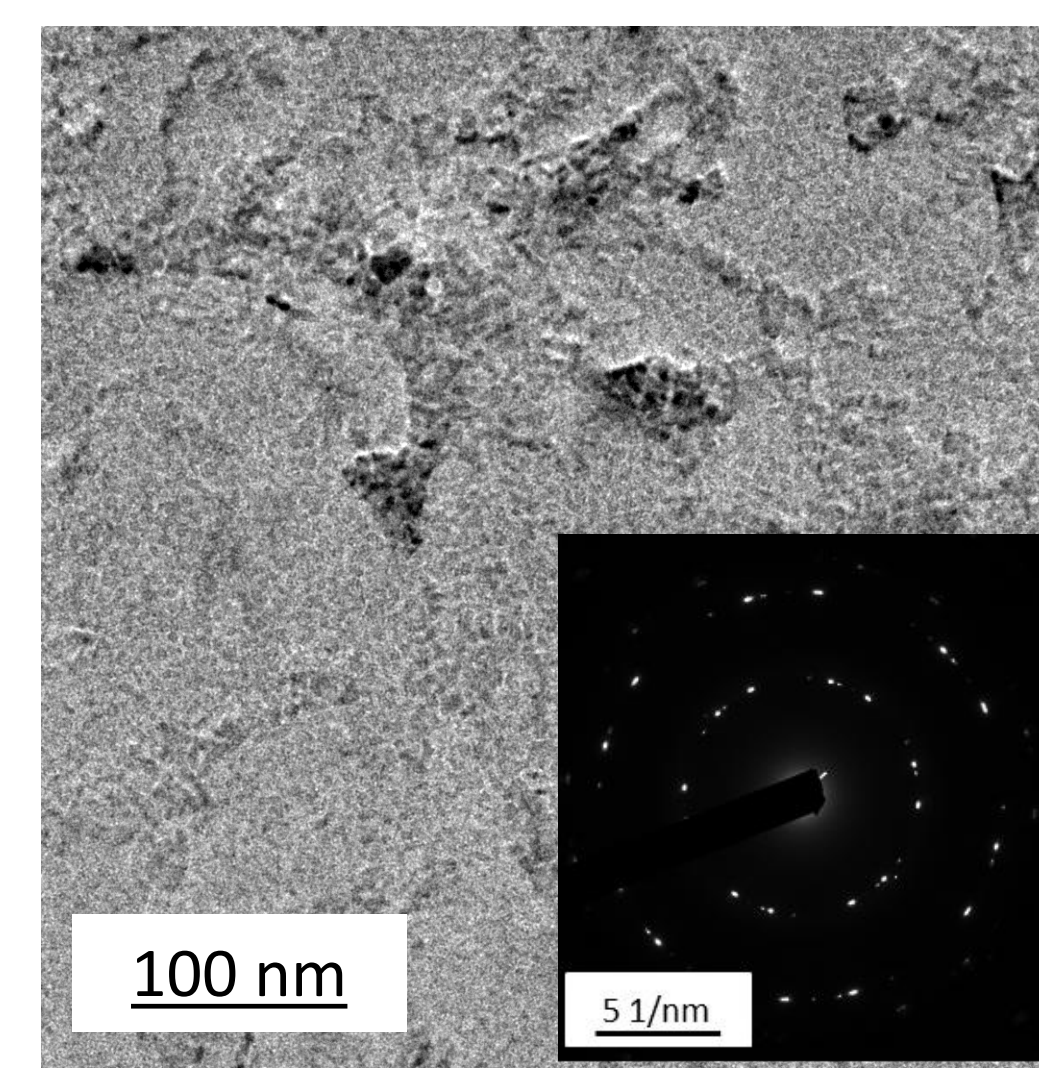
## Structural characterization

### X-Ray Diffraction (XRD) Co K $\alpha$ ( $\lambda = 1.7902 \text{ \AA}$ ) Scanning Electron Microscopy (SEM)

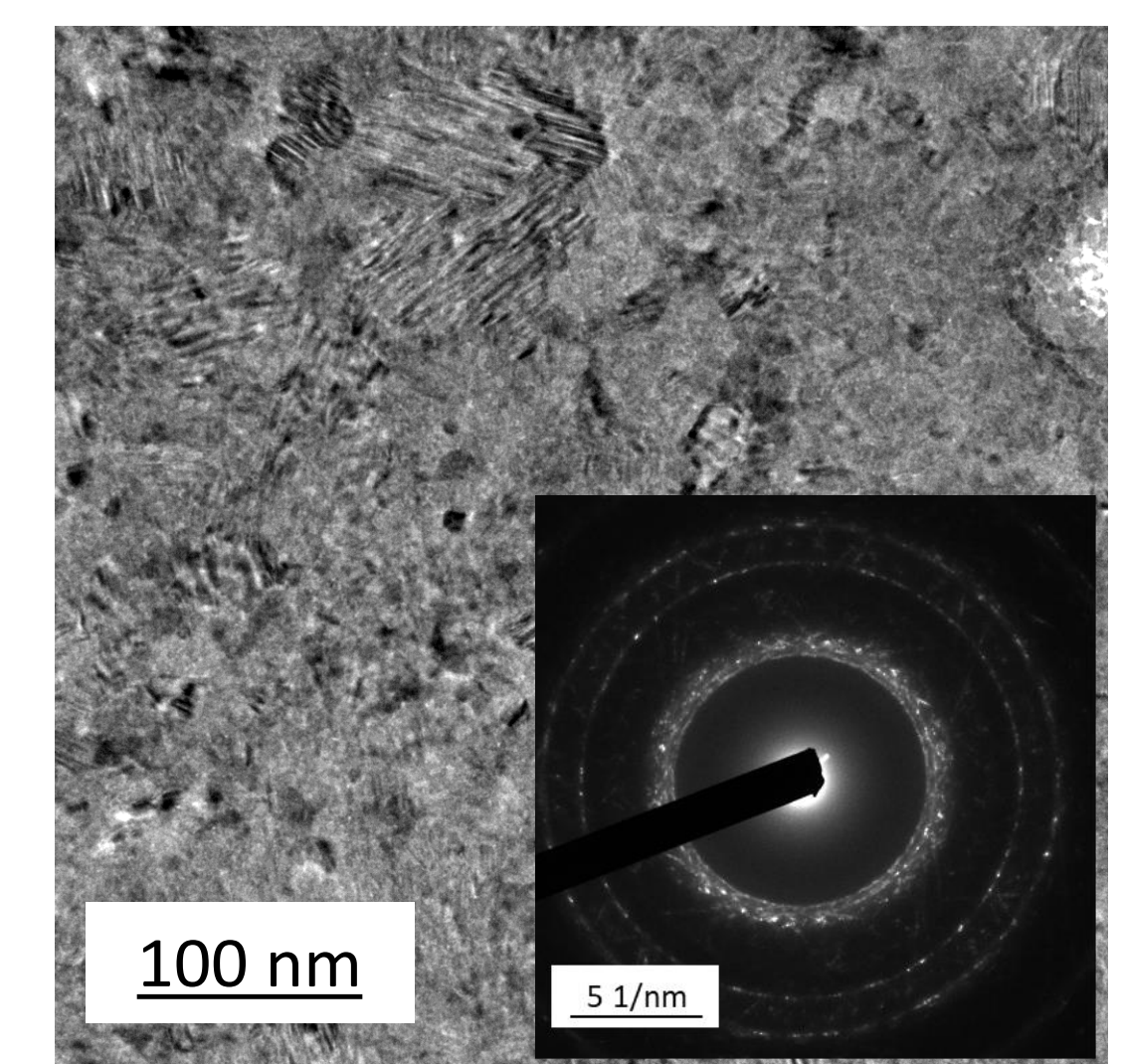


CoCrNi (630 nm)

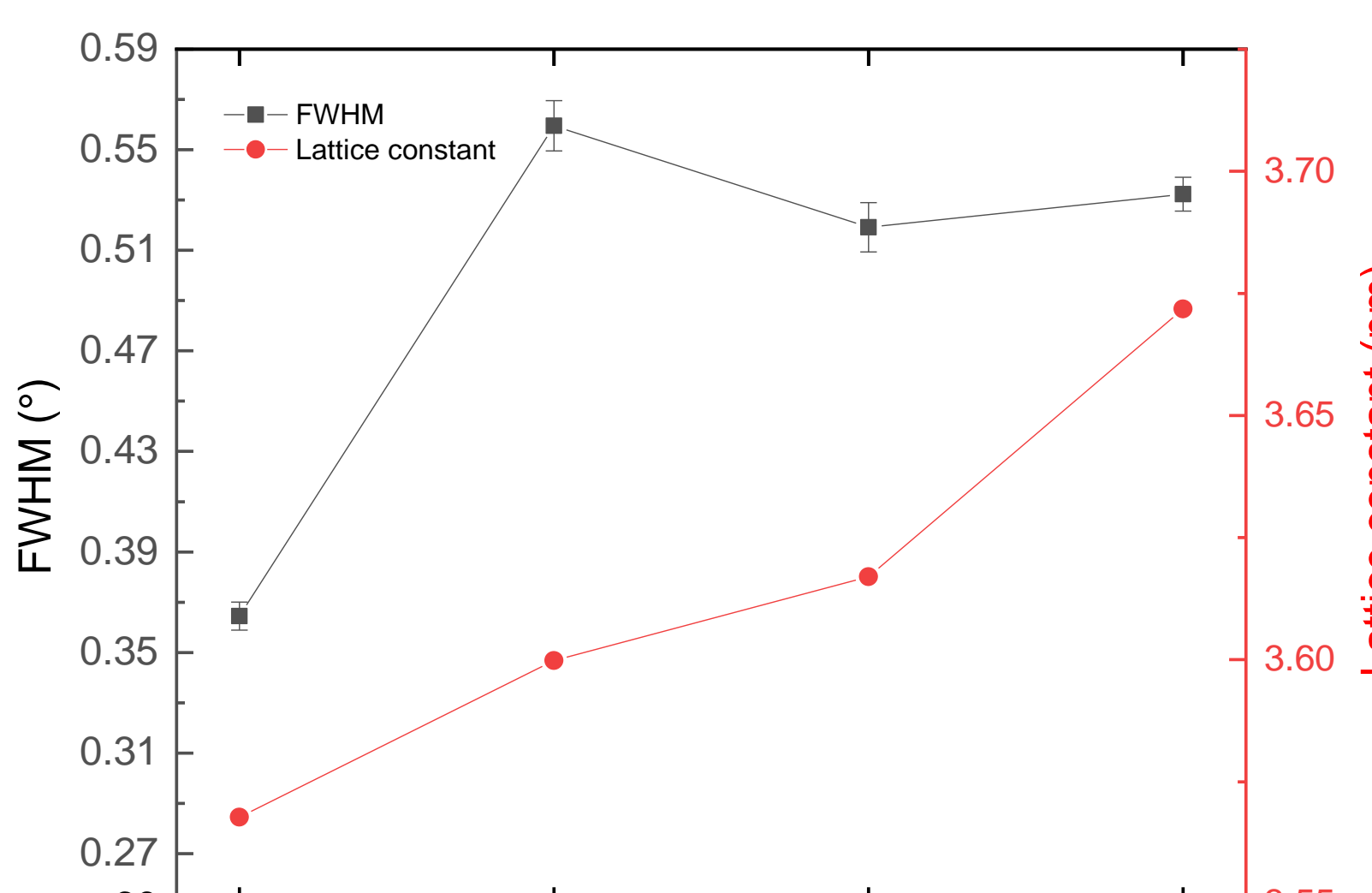
### Transmission Electron Microscopy (TEM)



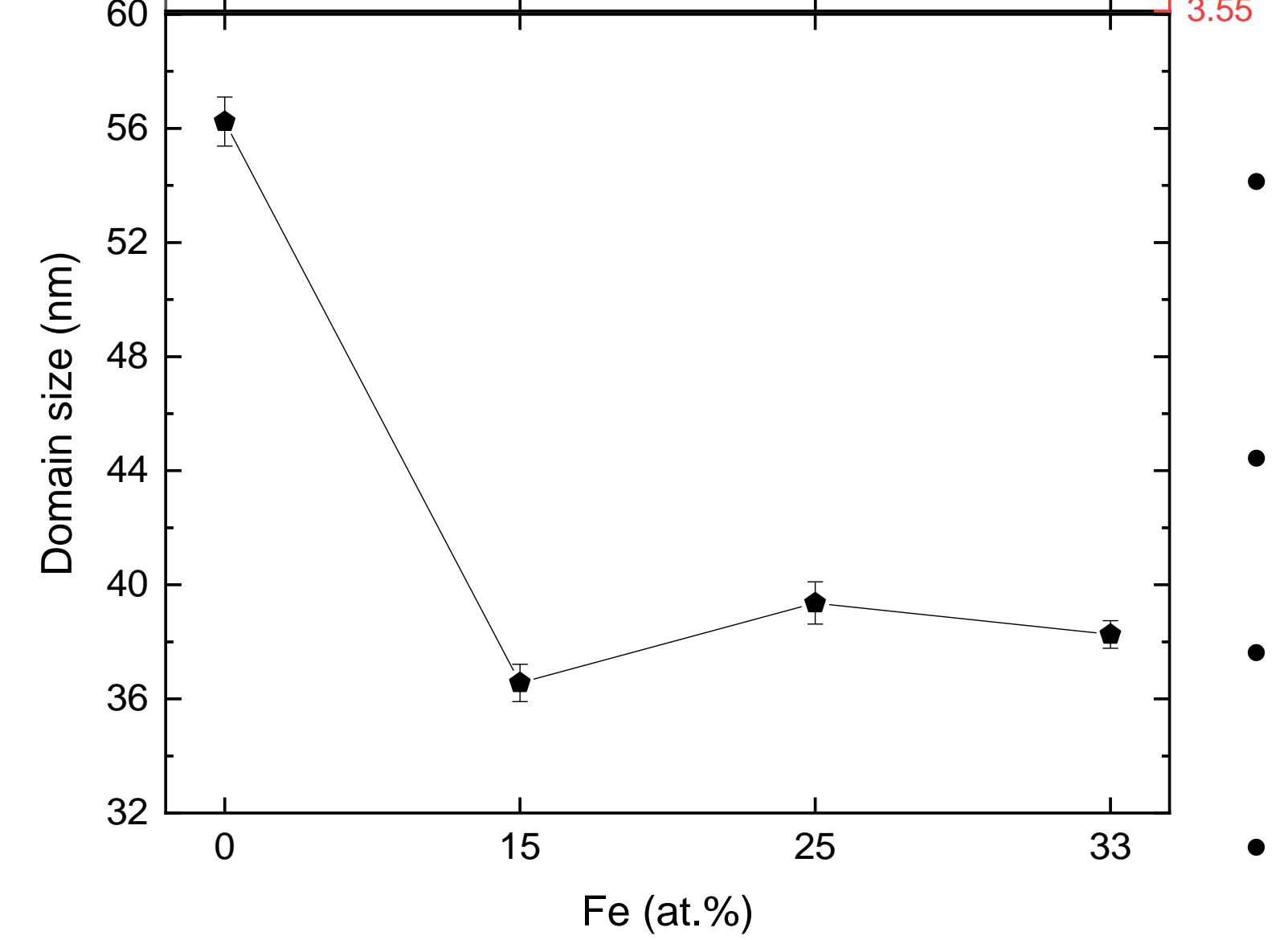
CoCrNi (15 nm)



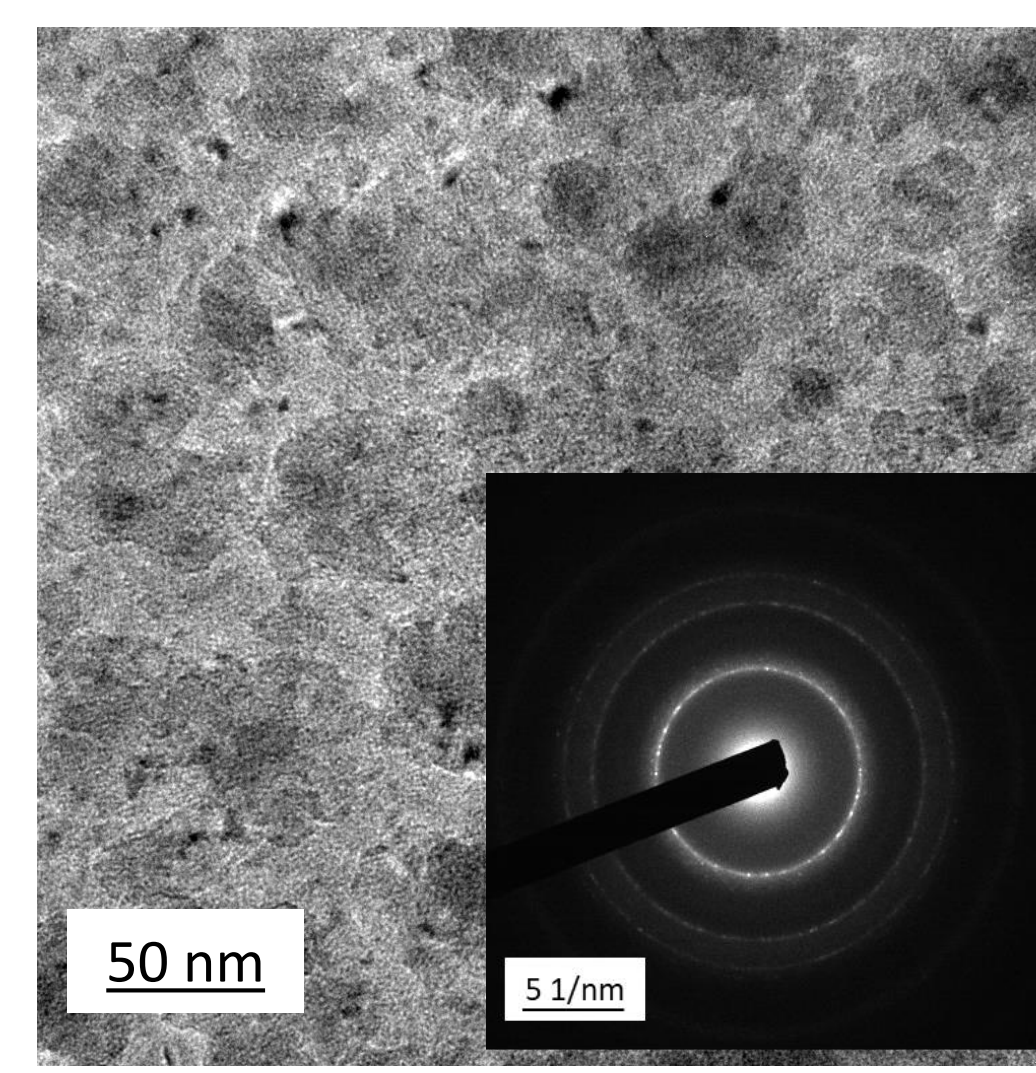
CoCrNi (40 nm)



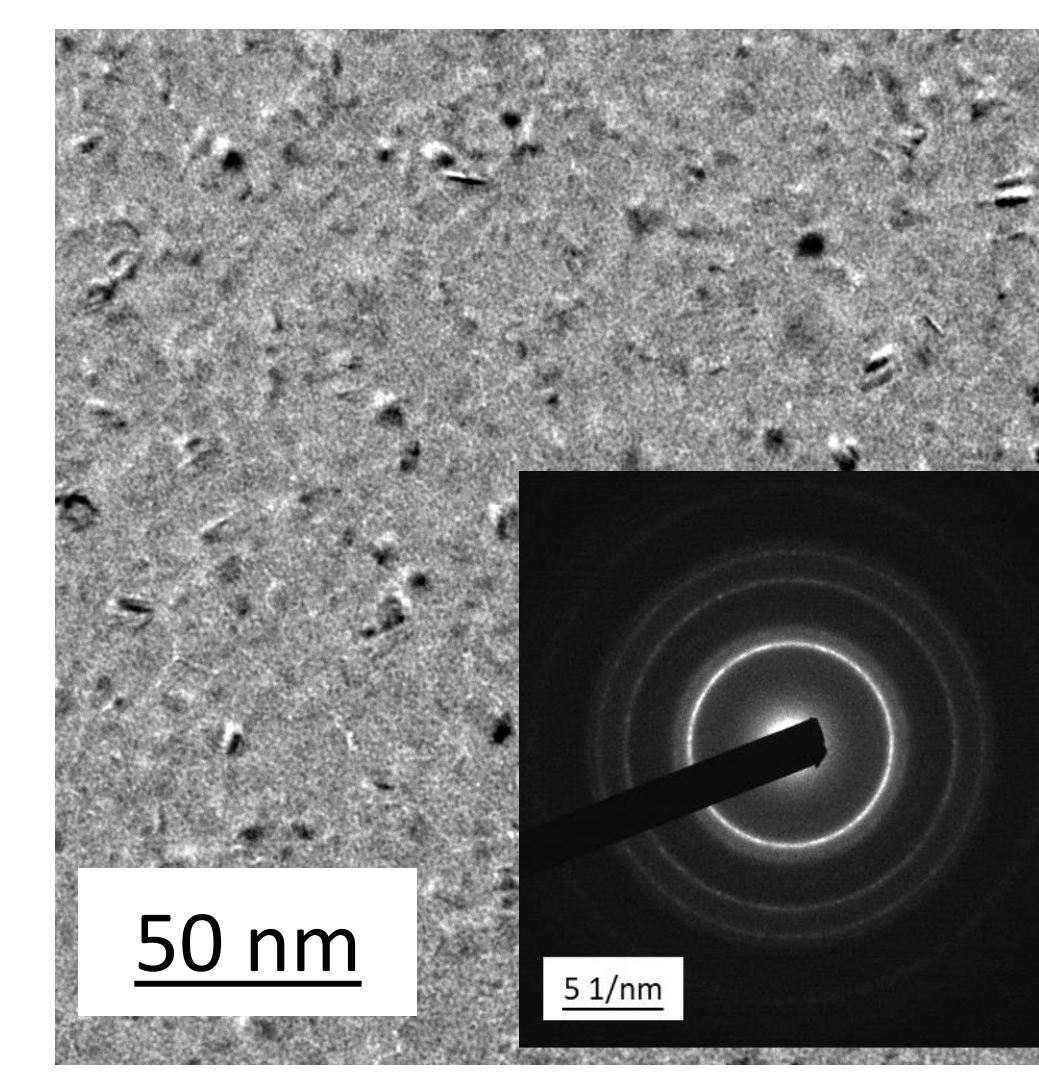
- CoCrNi  $\rightarrow$  Sharp FCC (111) peak
- Increase in Fe content  $\rightarrow$  FCC (111) peak shift to smaller angles, lattice constant increases
- Fe being the 2<sup>nd</sup> largest of these elements (atomic radius 156 pm) can enhance lattice distortion



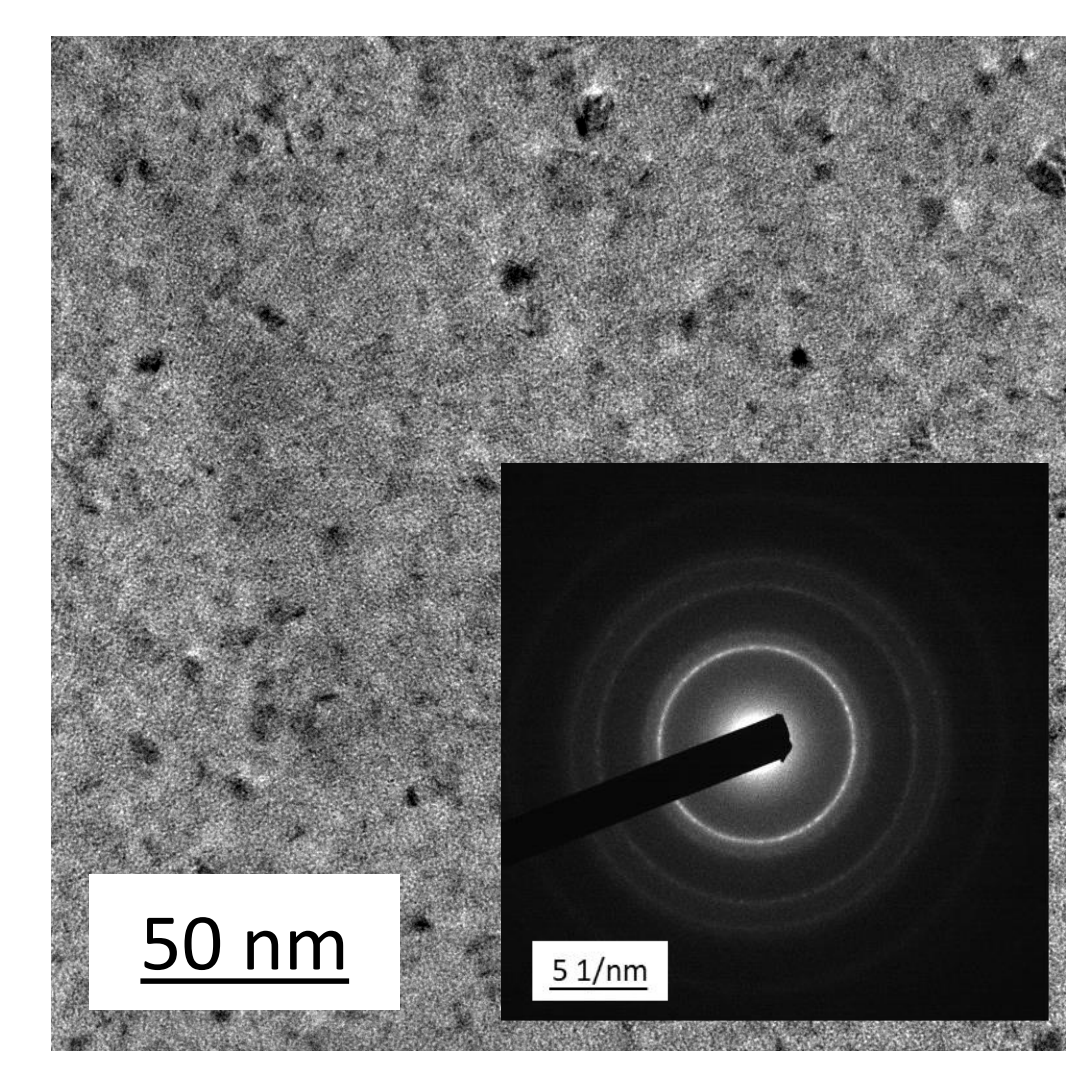
- HCP CoCrNi (15 nm)  $\rightarrow$  FCC/amorphous CoCrNi (40 nm)
- Possible appearance of stacking faults
- Nanocrystalline  $\text{Fe}_x(\text{CoCrNi})_{1-x}$  preferred FCC (111) orientation
- Addition of Fe results in smaller crystallite sizes



$\text{Fe}_{0.15}(\text{CoCrNi})_{0.85}$



$\text{Fe}_{0.25}(\text{CoCrNi})_{0.75}$



$\text{Fe}_{0.33}(\text{CoCrNi})_{0.67}$

## Conclusion

- FCC (111) phase was confirmed with Fe addition up to 33 at.%
- The increase in E and H can be attributed to the small domain size (<20 nm) and the sharp FCC (111) texture
- Planar defects like Stacking Faults (SFs) and nanotwins may also be responsible for enhancing mechanical properties
- Ductile behaviour of CoCrNi on Kapton<sup>®</sup> polymer can be enhanced with Fe

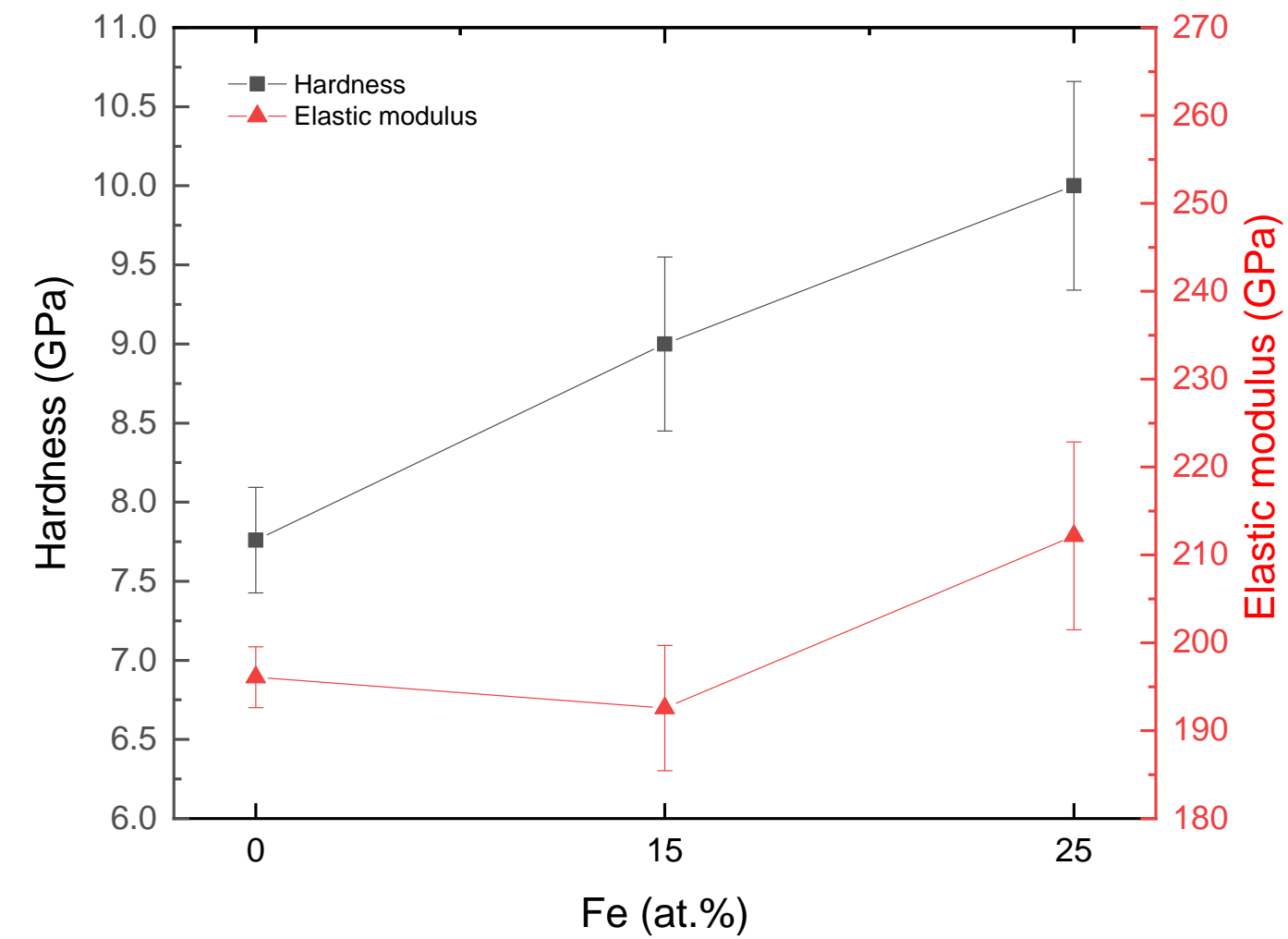
## Prospectives

- Deeper exploration of structural and mechanical properties
- Investigating microscale mechanical properties with in-situ SEM techniques



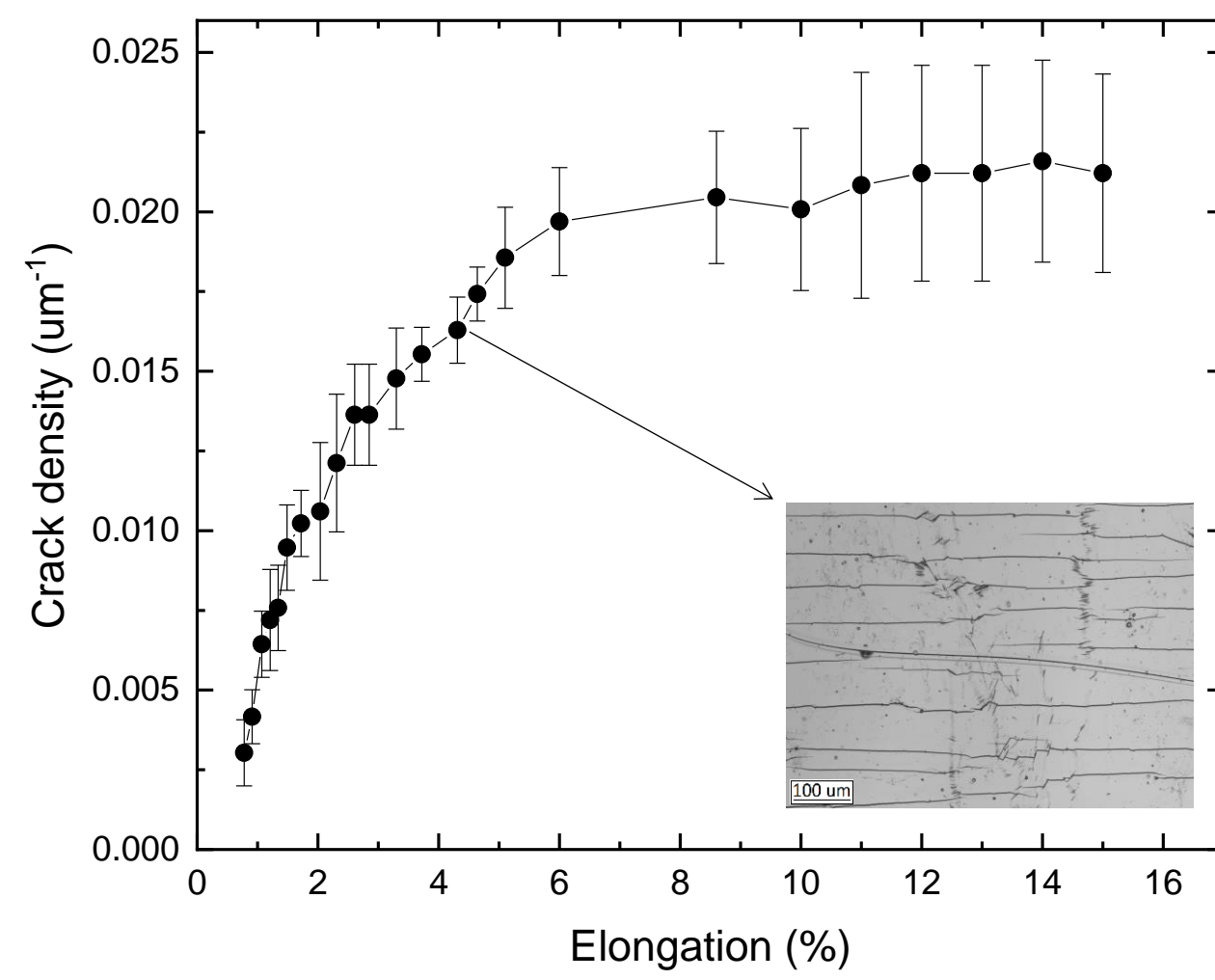
## Mechanical characterization

### Nanoindentation



Fe (at.%) 0  $\rightarrow$  25

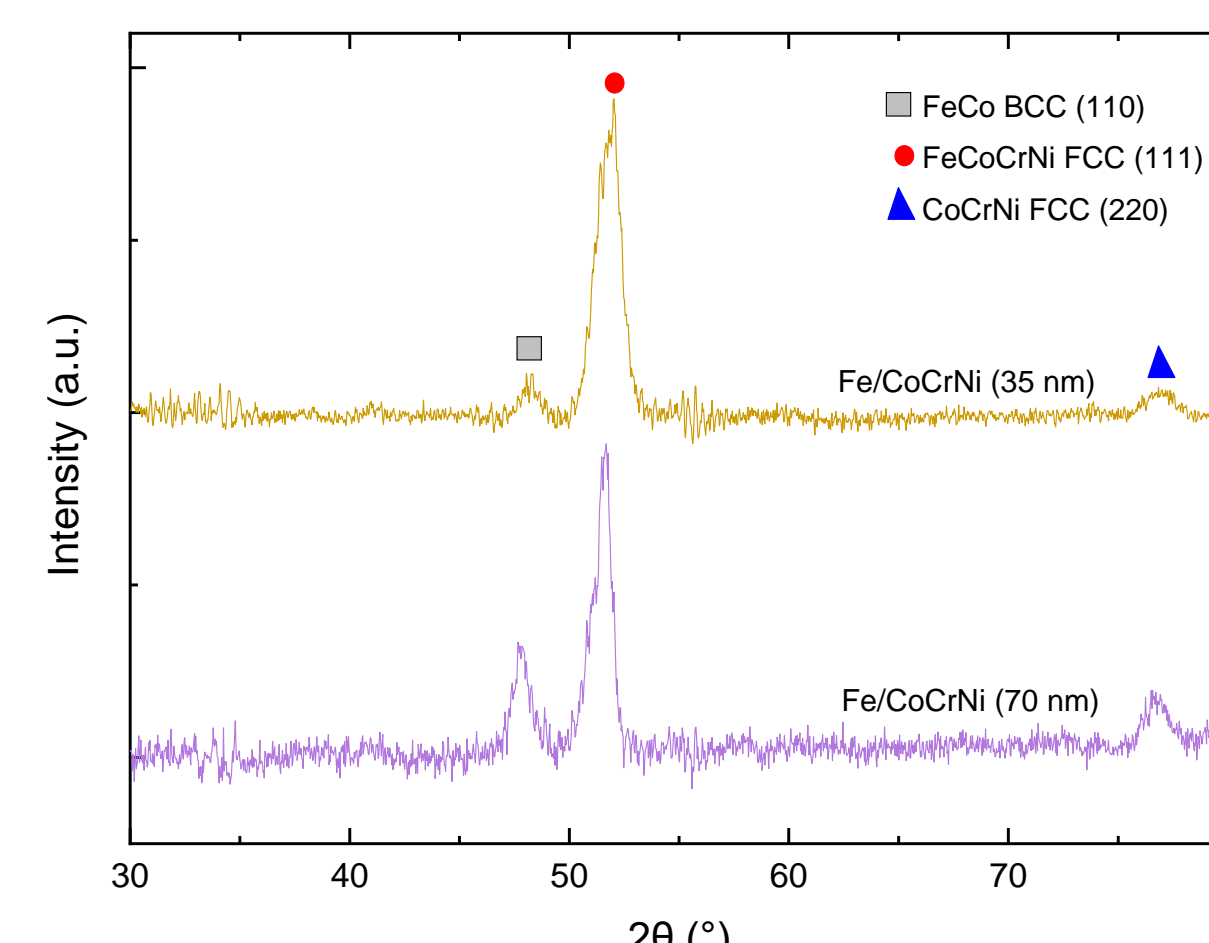
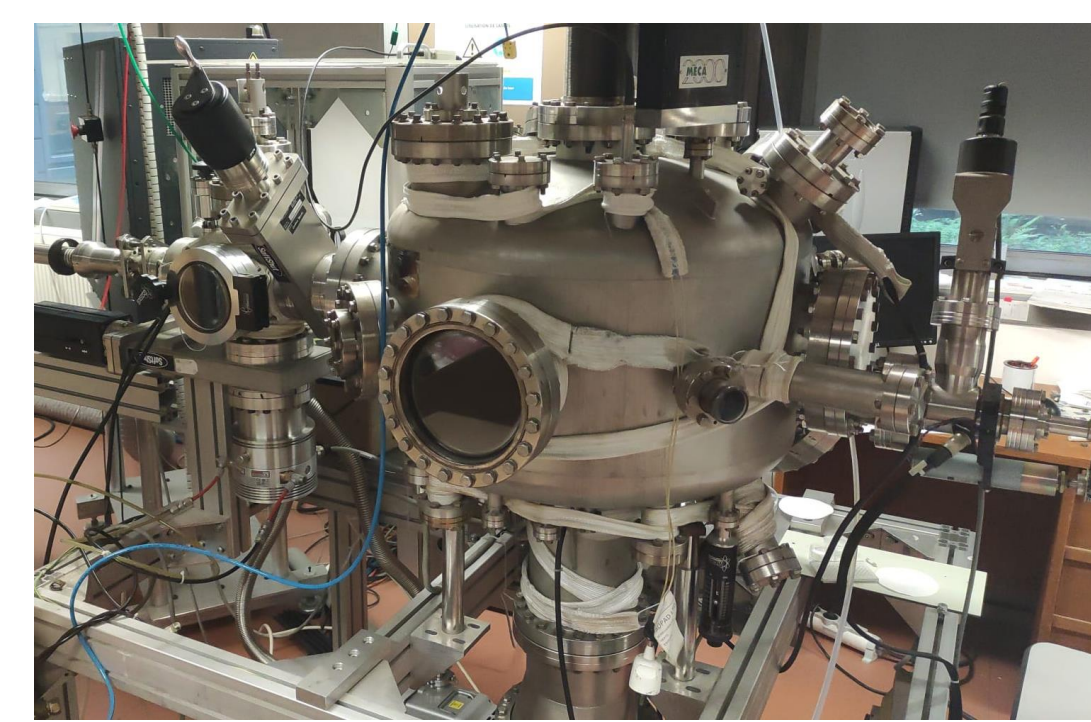
### Tensile test



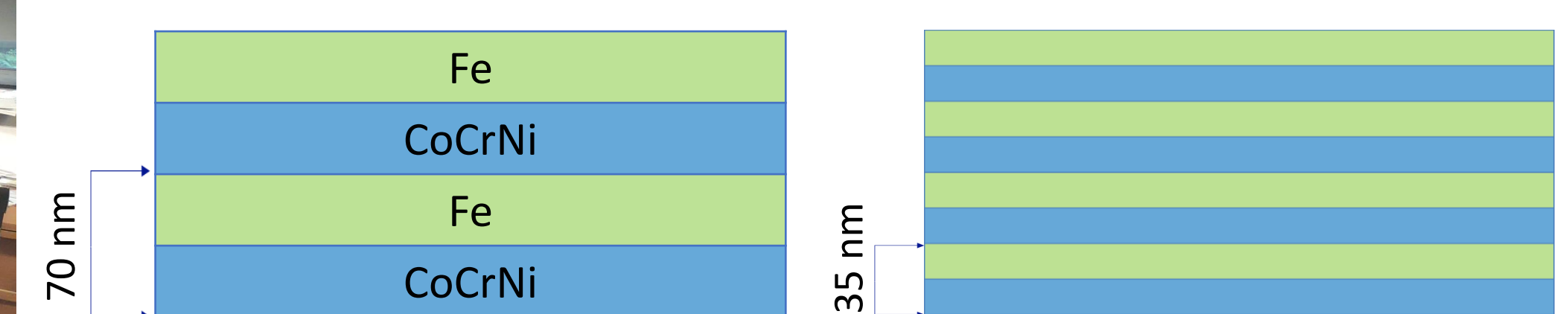
CoCrNi

- E, H increase up to 210 GPa, 10 GPa
- Presence of Stacking Faults, nanotwins
- Tested on Kapton<sup>®</sup> polymer
- Cracks initiate at 0.78%, buckling at 4.31%

## Pulsed Laser Deposition



### Multilayers of Fe/CoCrNi



Total thickness: 700 nm

### Characterization of Fe/CoCrNi multilayers

- Observation of FeCo BCC (110) and CoCrNi FCC (220) peaks
- Effects of reducing bi-layer period (FWHM increases, Domain size decreases down to 8 nm, H enhanced up to 10 GPa)

- Pulsed laser deposition (PLD)  $\rightarrow$  synthesis of multicomponent nanoparticles and investigation of film growth regime.
- $\text{Pd}_x(\text{CoCrNi})_{1-x}$  can be studied and compared to  $\text{Fe}_x(\text{CoCrNi})_{1-x}$  thin films

## References

- [1] F. Cao, *et al.*, Surface and Coatings Technology, Volume **335**, 2018
- [2] M.K. Kini *et al.*, Surface & Coatings Technology **410** (2021)
- [3] P. Nagy, *et al.*, Materials **2022**, 15, 2319

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