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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

Arjun Bharath Curam^{1,2*}, Vianney Mille¹, Gregory Abadias³, Philippe Djemia¹, Christian Ricolleau², Matteo Ghidelli¹

¹LSPM-CNRS, Laboratoire des Sciences des Procédés et des Matériaux, Université Sorbonne Paris Nord

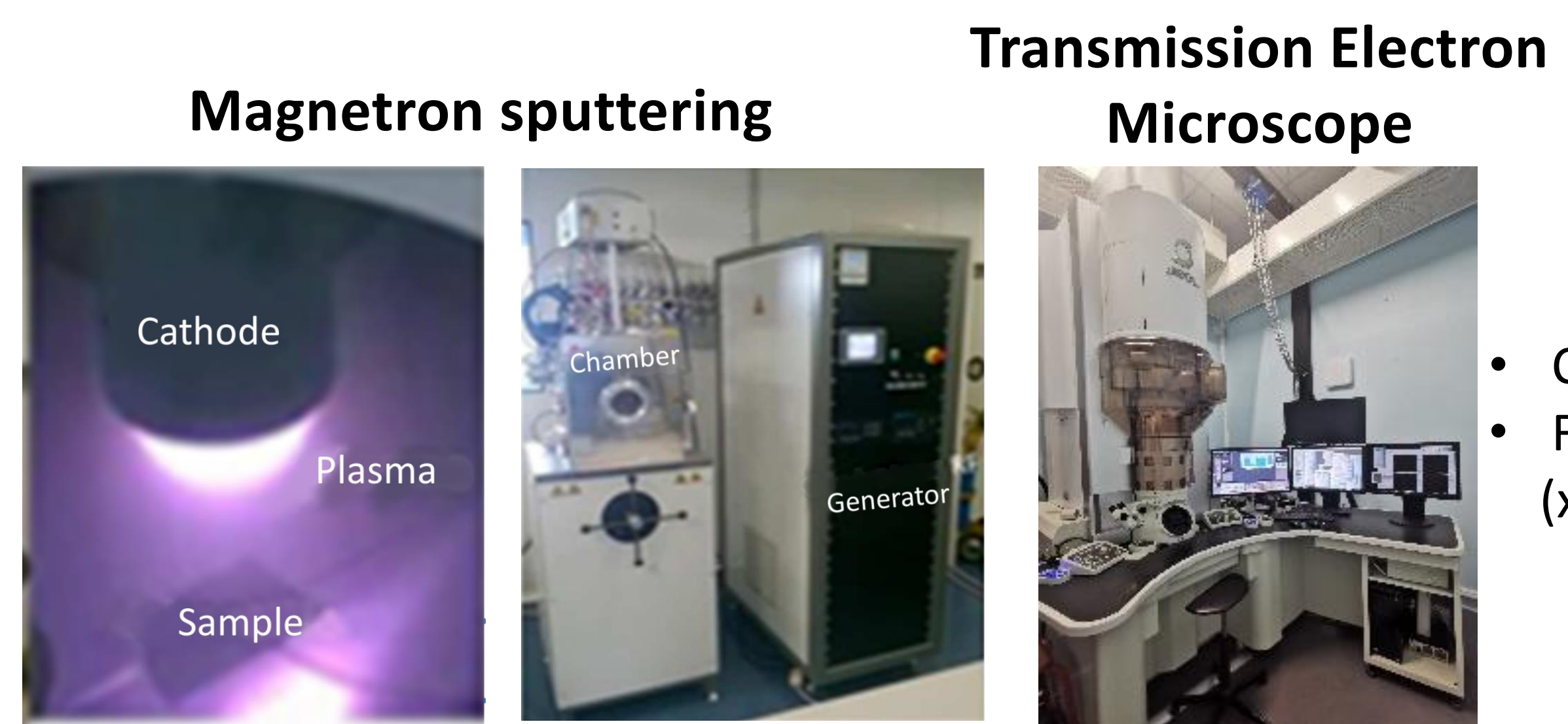
²MPQ, Laboratoire Matériaux Et Phénomènes Quantiques, Université Paris Cité

³Institut Pprime-CNRS, Université de Poitiers-ISA ENSMA

* arjun.curam@lspm.cnrs.fr

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 σ 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.

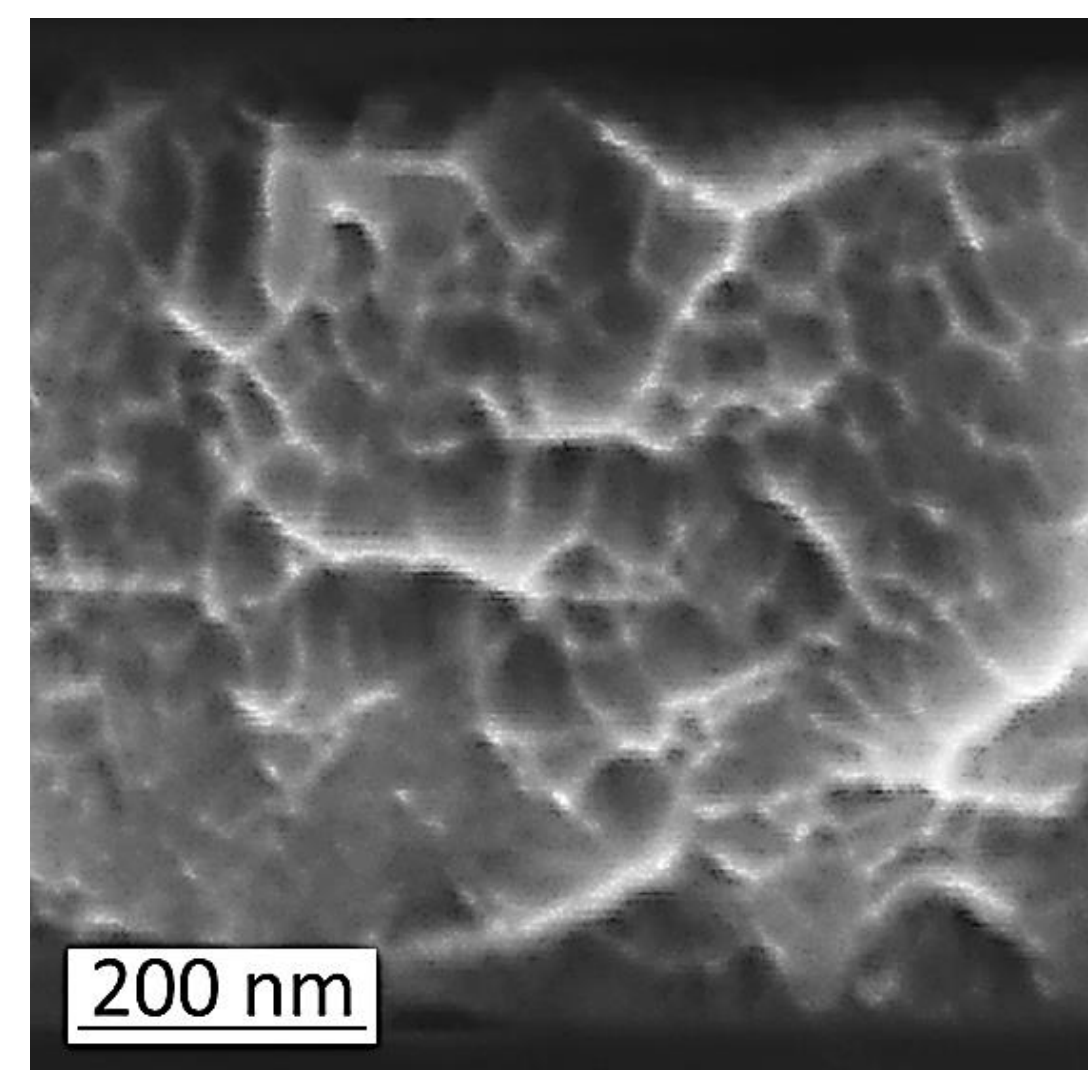
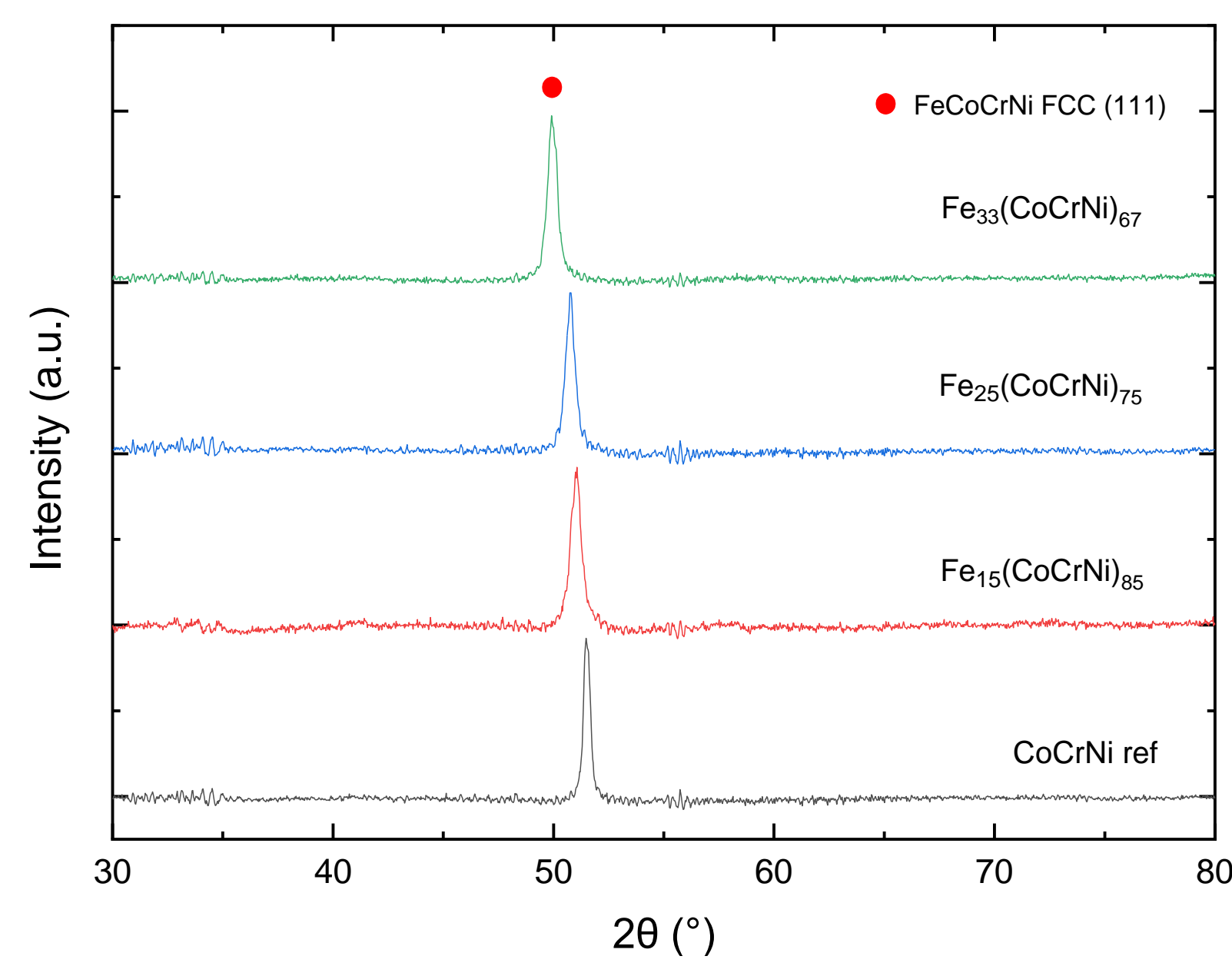


Compositions

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

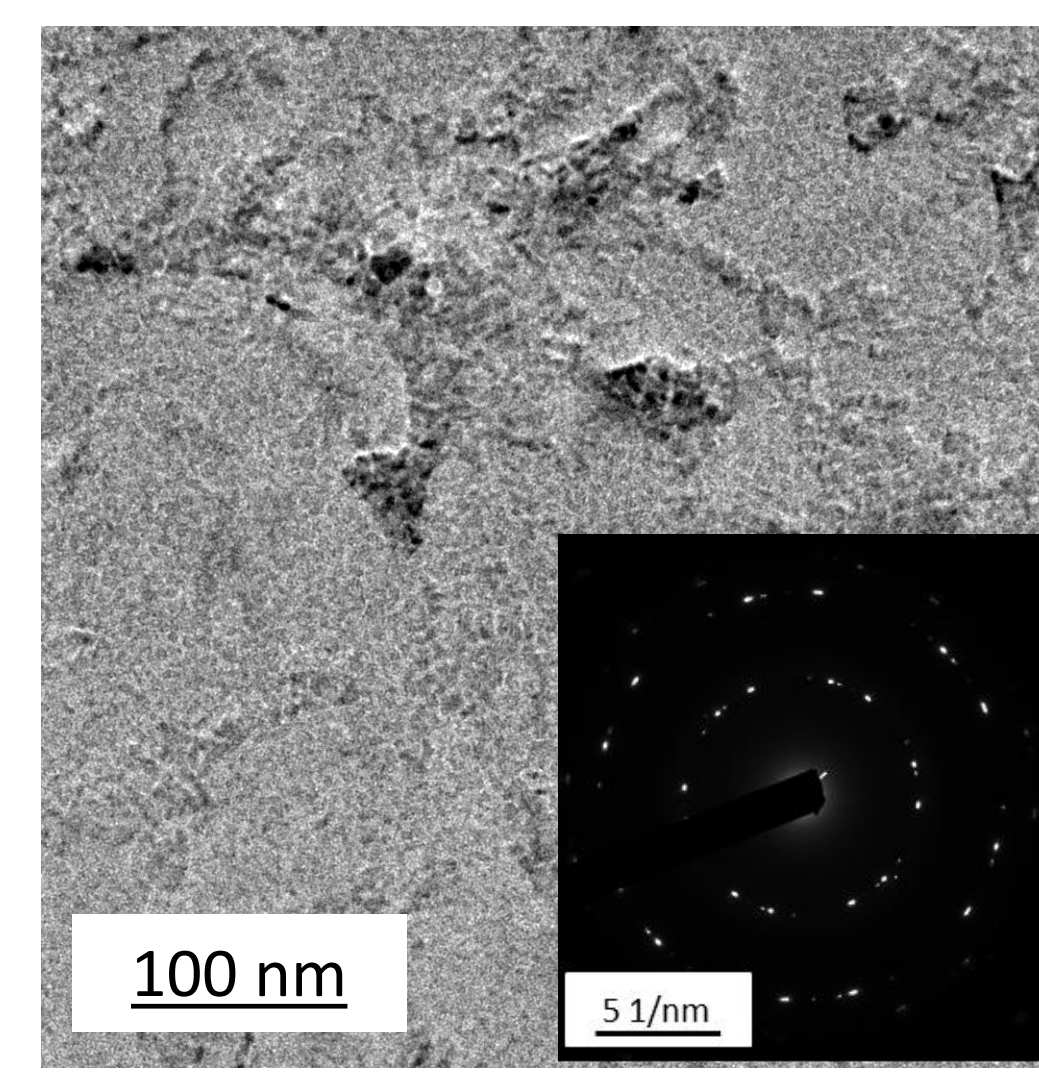
Structural characterization

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

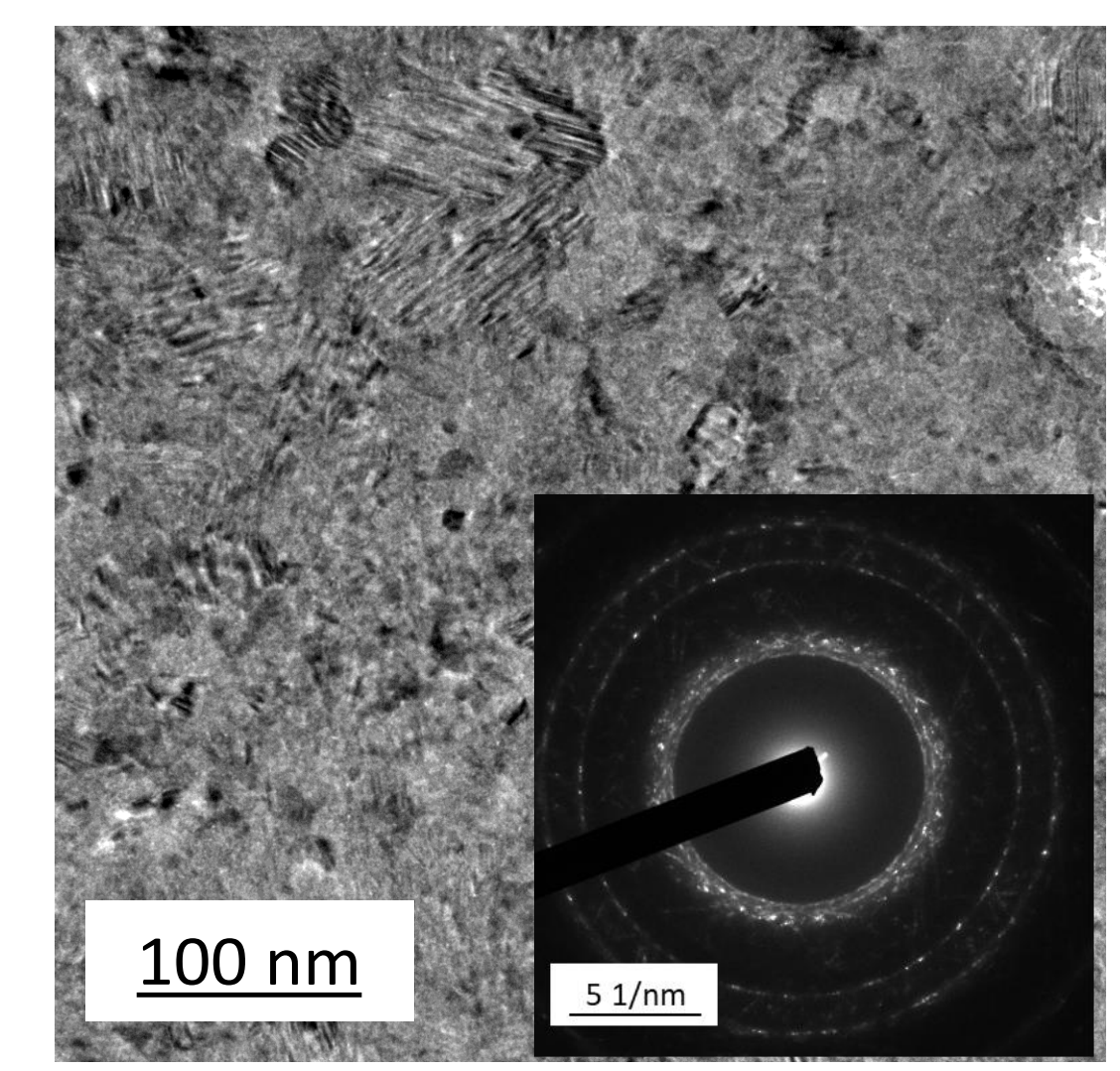


CoCrNi (630 nm)

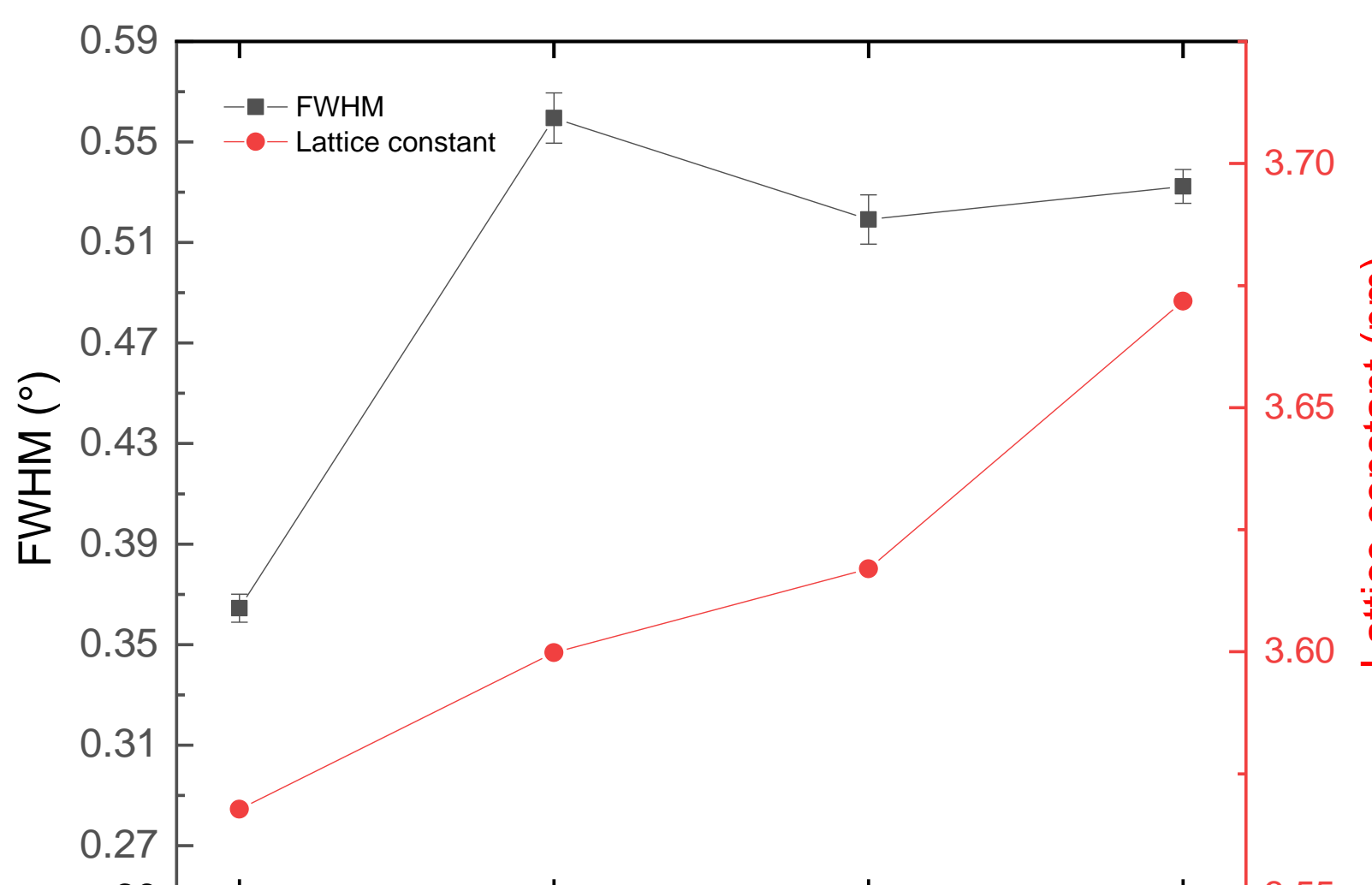
Transmission Electron Microscopy (TEM)



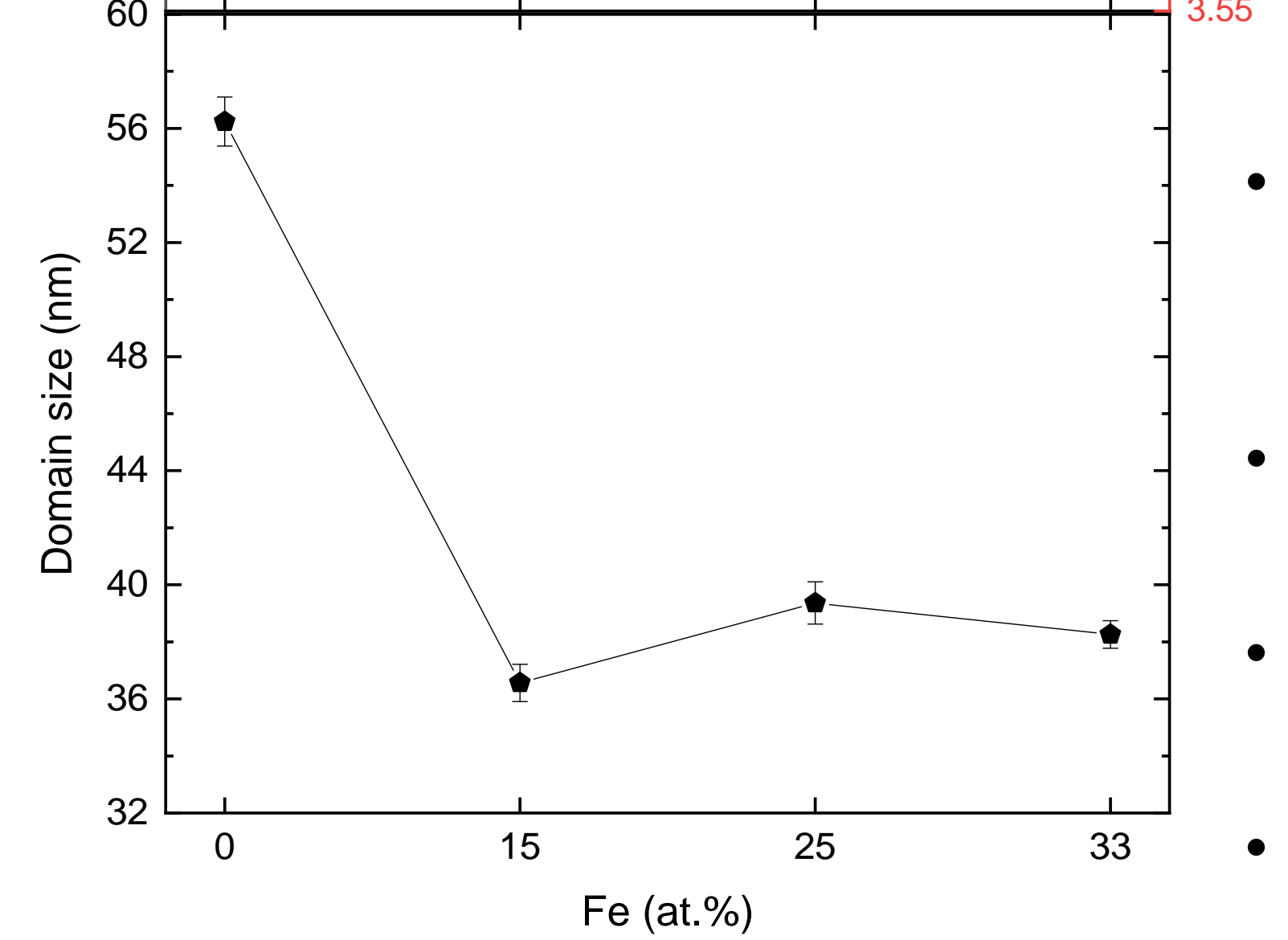
CoCrNi (15 nm)



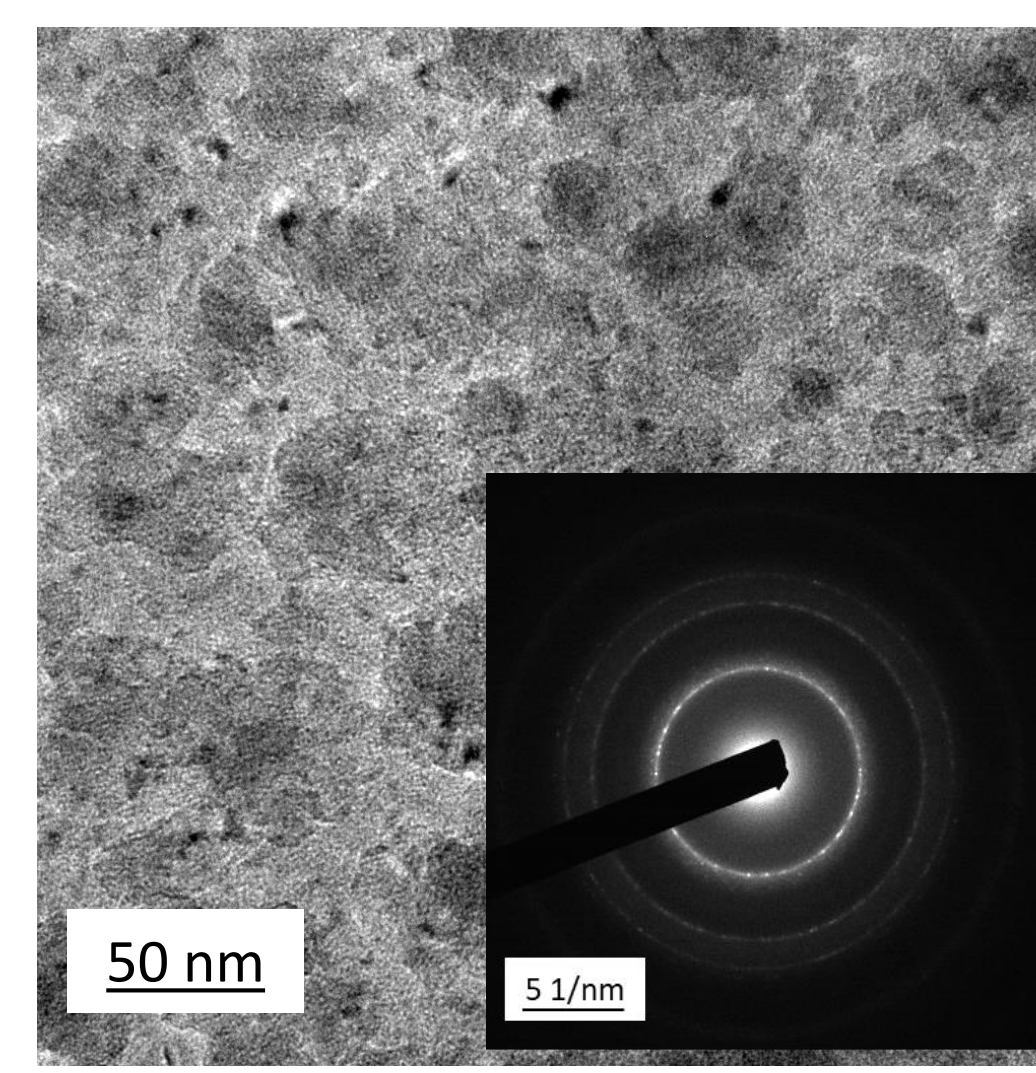
CoCrNi (40 nm)



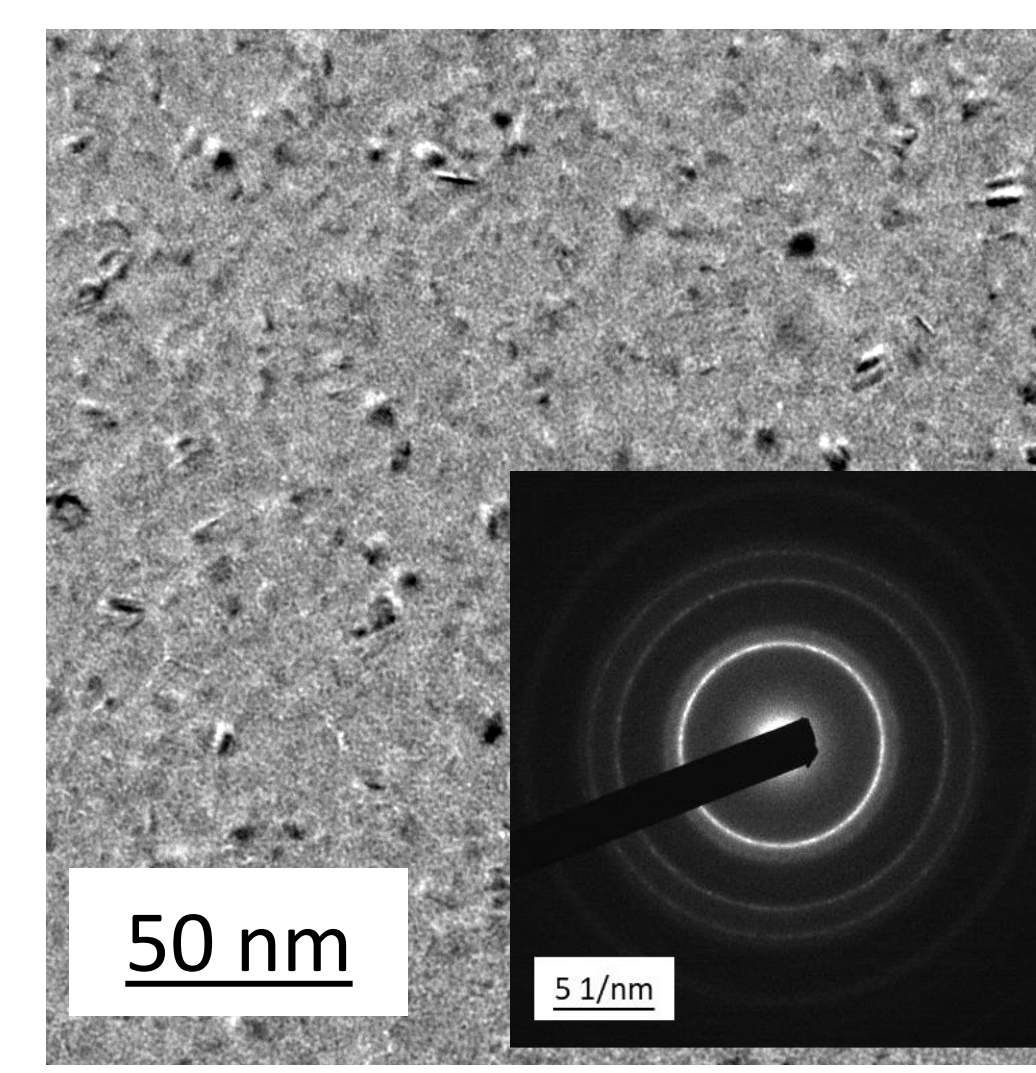
- CoCrNi \rightarrow Sharp FCC (111) peak
- Increase in Fe content \rightarrow FCC (111) peak shift to smaller angles, lattice constant increases
- Fe being the 2nd largest of these elements (atomic radius 156 pm) can enhance lattice distortion



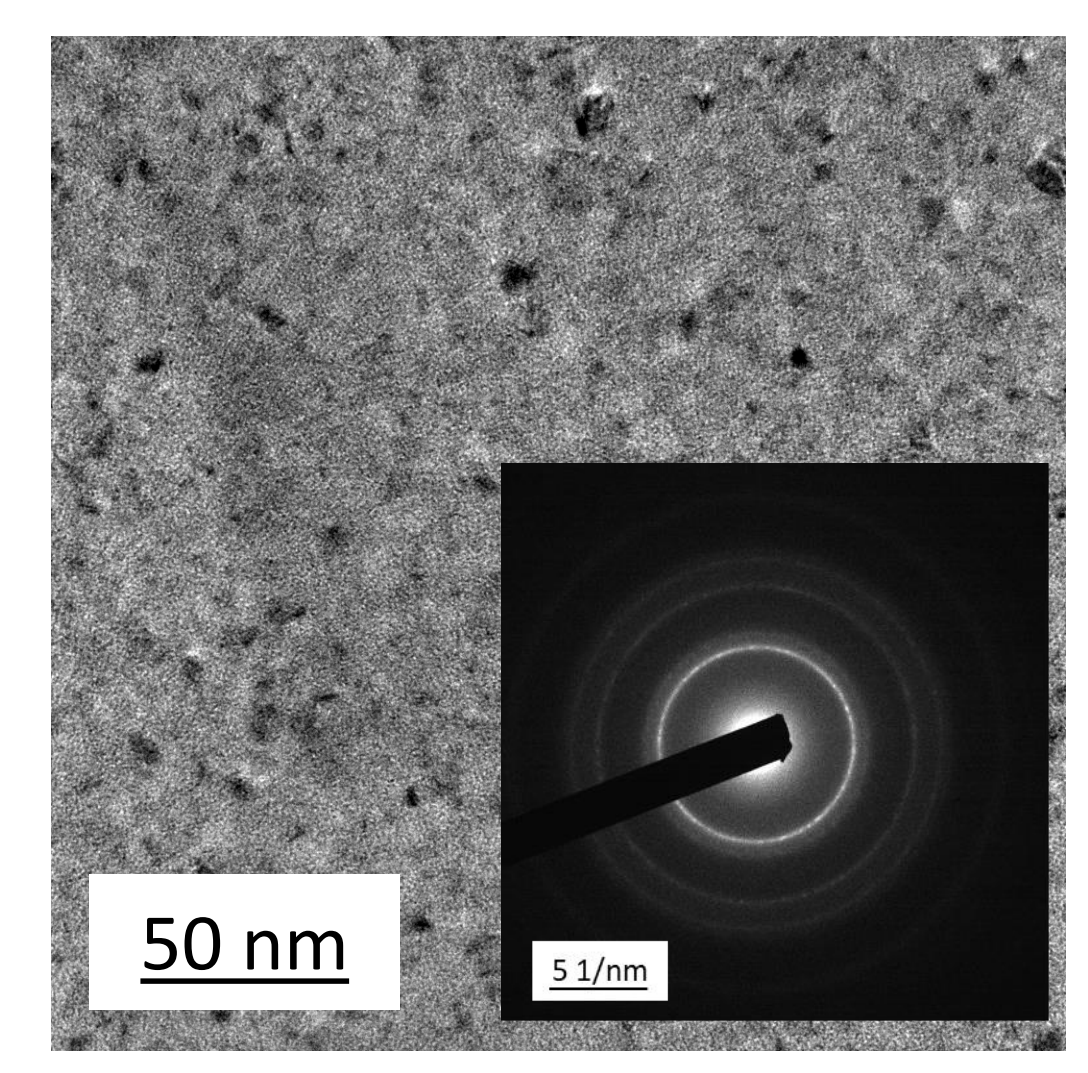
- HCP CoCrNi (15 nm) \rightarrow FCC/amorphous CoCrNi (40 nm)
- Possible appearance of stacking faults
- Nanocrystalline $\text{Fe}_x(\text{CoCrNi})_{1-x}$ preferred FCC (111) orientation
- Addition of Fe results in smaller crystallite sizes



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



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



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

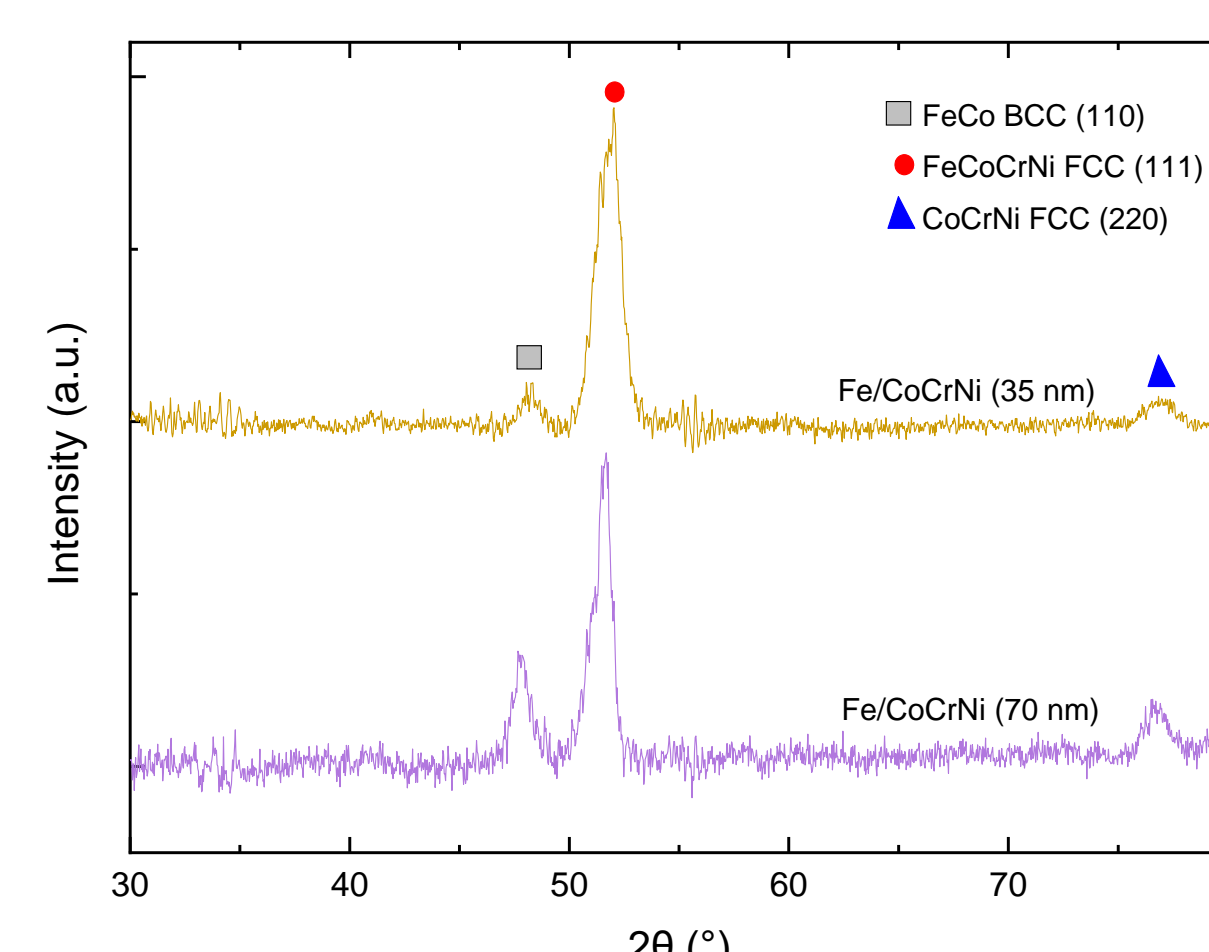
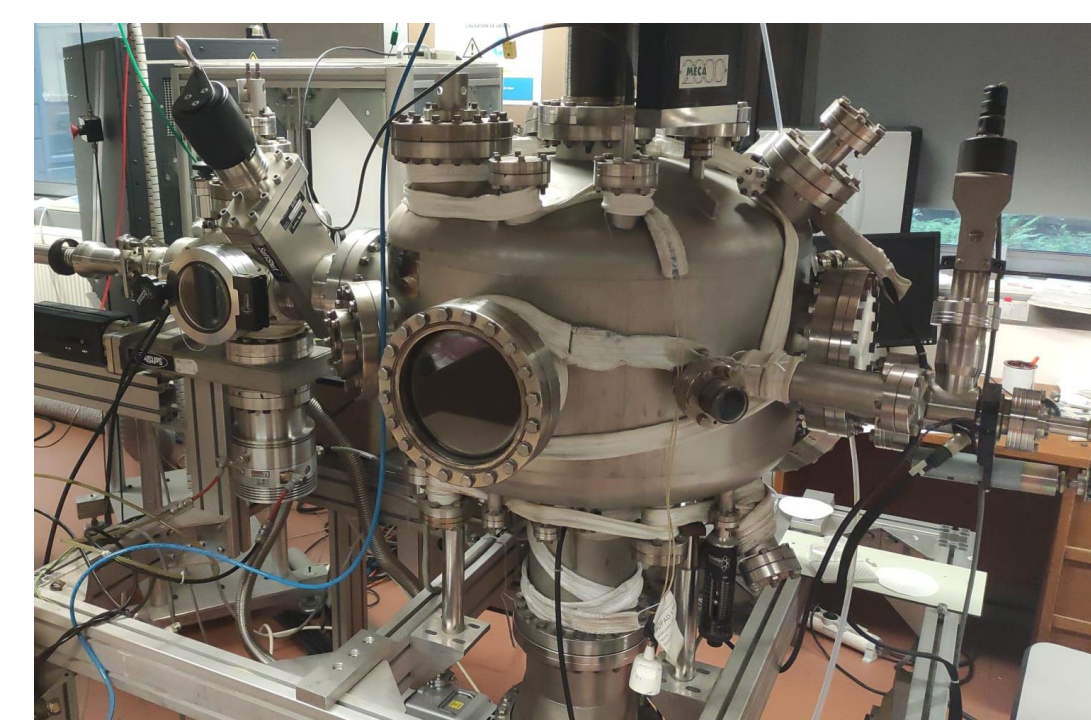
Conclusion

- 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[®] polymer can be enhanced with Fe

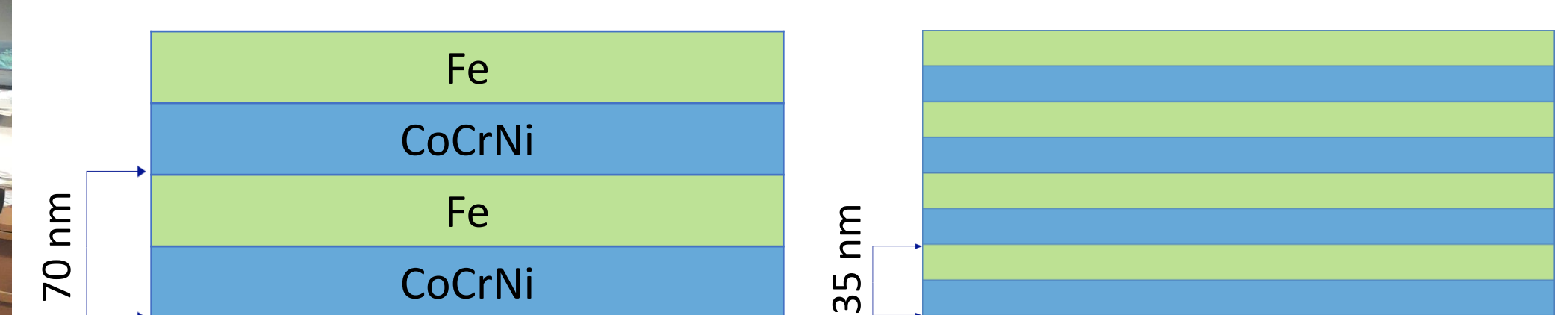
Prospectives

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

Pulsed Laser Deposition



Multilayers of Fe/CoCrNi

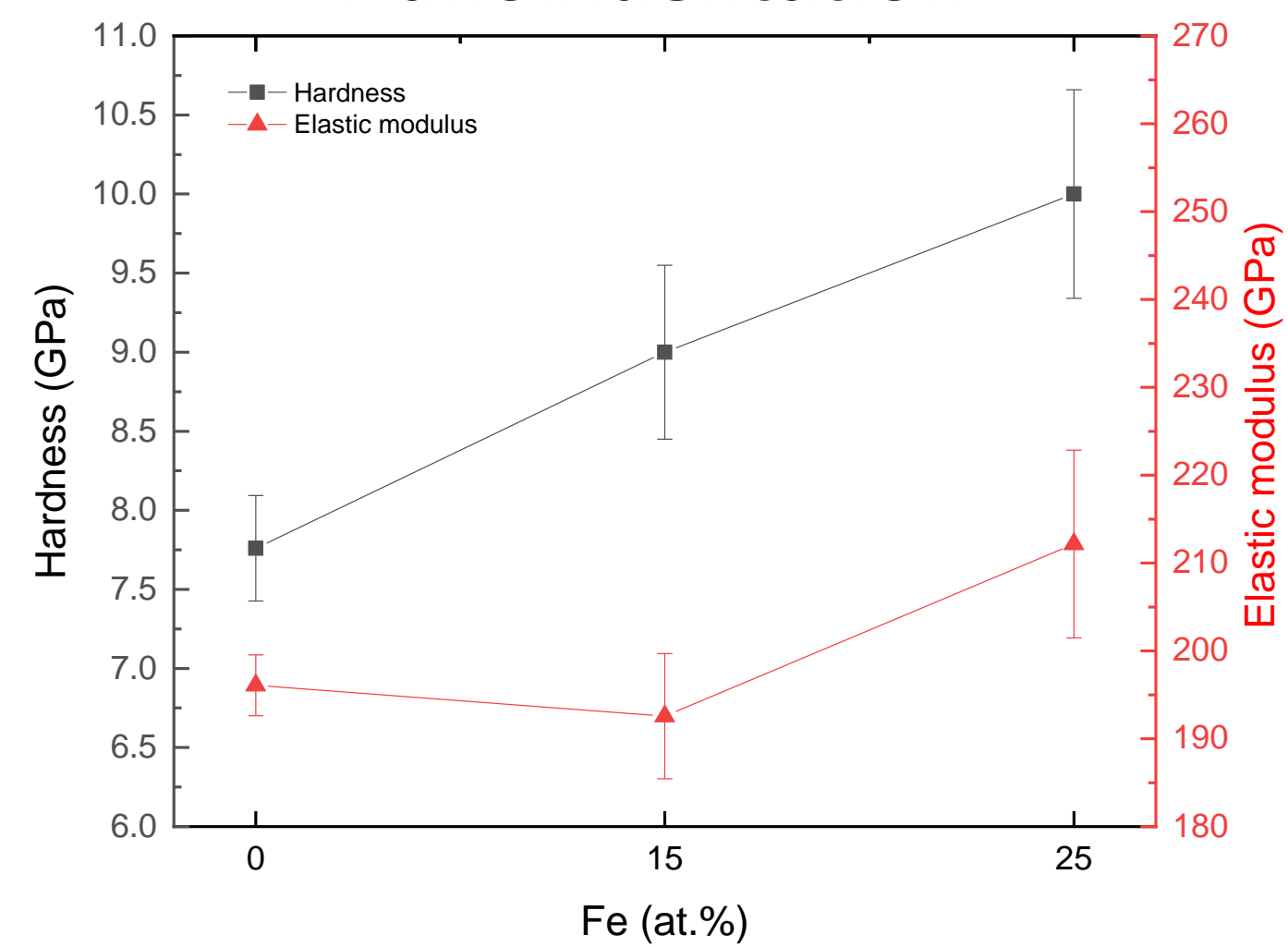


Characterization of Fe/CoCrNi multilayers

- 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)

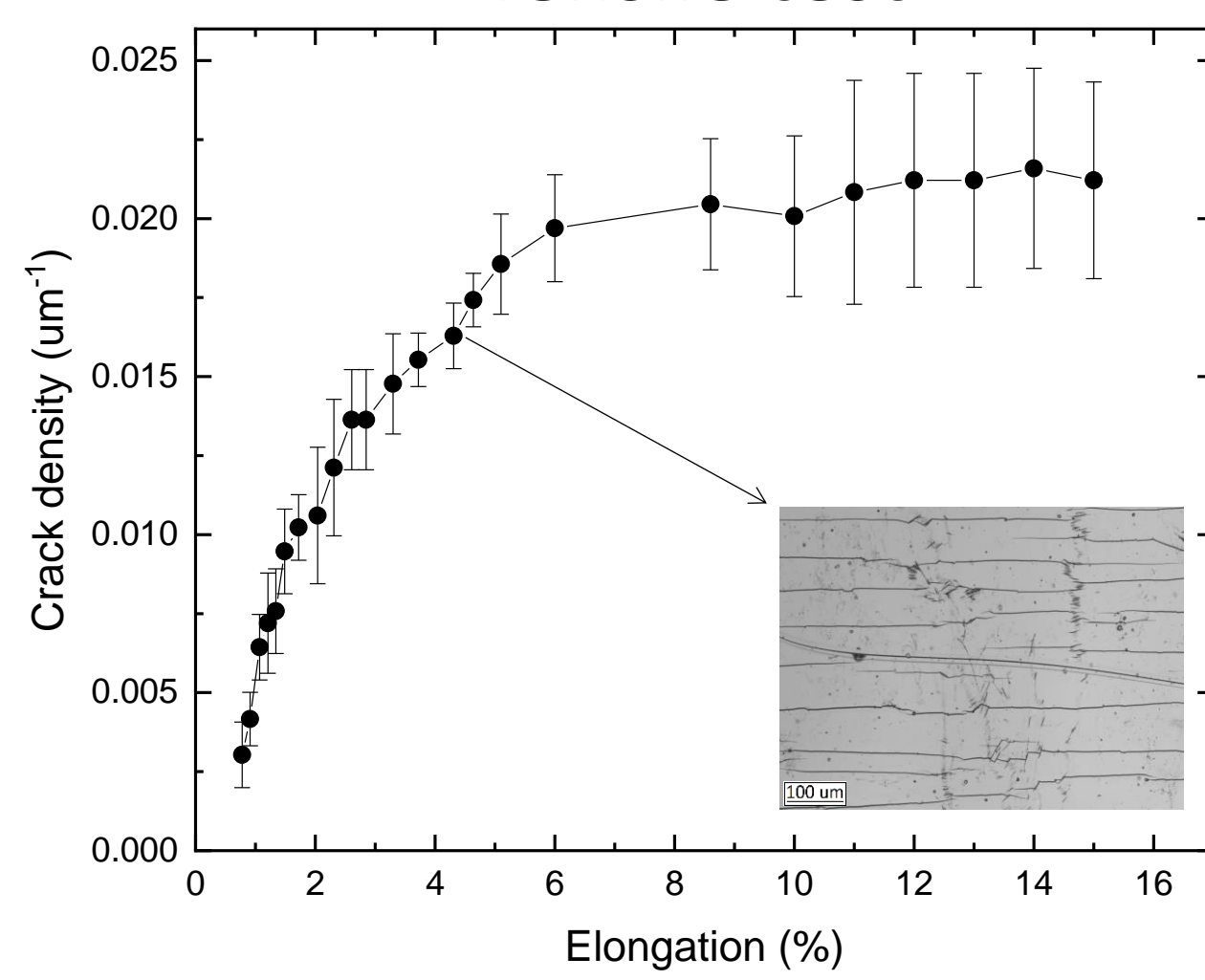
Mechanical characterization

Nanoindentation



Fe (at.%) 0 \rightarrow 25

Tensile test



CoCrNi

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

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

Acknowledgements

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