

Tatiana Kormilina¹, Georg Haberfehlner², Thomas Radlinger², Gerald Kothleitner^{1,2}

1. Graz Center for Electron Microscopy (ZFE), Steyrergasse 17, 8010 Graz, Austria
2. Institute of Electron Microscopy and Nanoanalysis (FELMI), Graz University of Technology, Steyrergasse 17, 8010 Graz, Austria

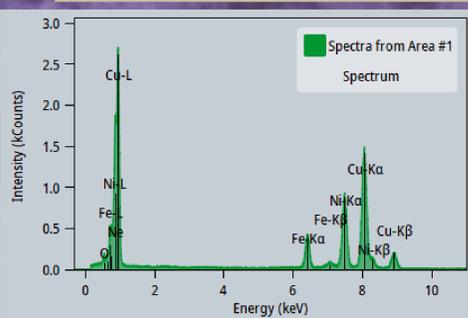
Introduction

Many properties and effects connected to the microstructure of materials are being redefined and expanded with the development of 3D characterization techniques. CuNiFe magnetic spinodal alloys are used here to demonstrate the capabilities of three-dimensional chemical analysis using analytical electron tomography techniques.

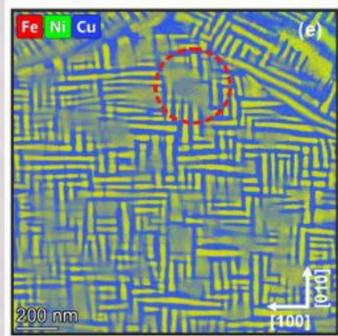
Main questions raised by 2D TEM investigations:

- Do the blurry areas (red circle) on elemental maps correspond to precipitate grains orthogonal to the field of view?
- Are the Ni+Fe -rich precipitates interconnected?

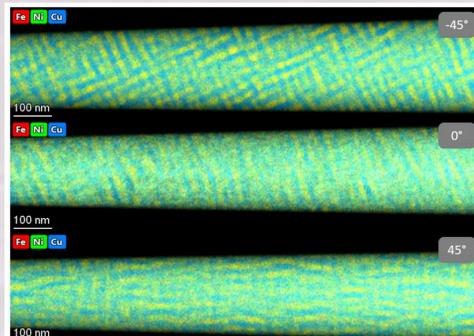
EDX



Typical EDX spectrum of CuNiFe summed over the area of a single projection

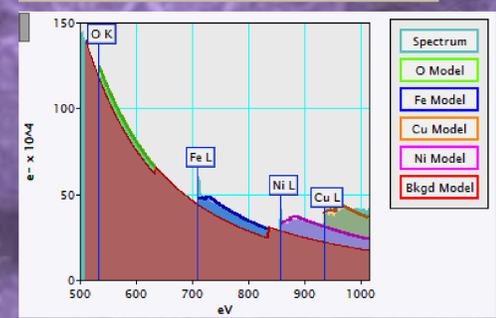


T. Radlinger, et al. *Journal of Alloys and Compounds* (2022): 166214.



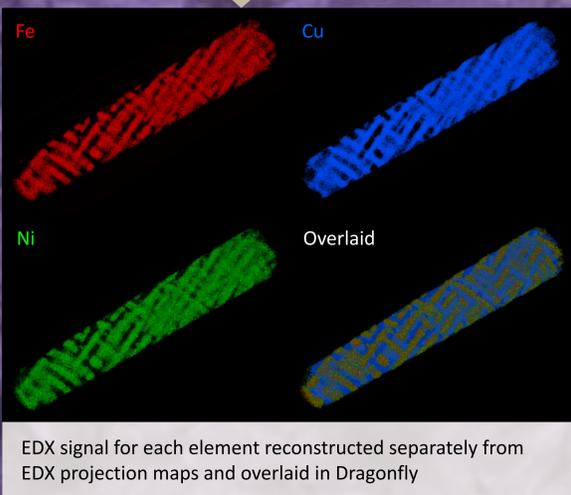
STEM EDX elemental maps taken at different tilt angles

EELS

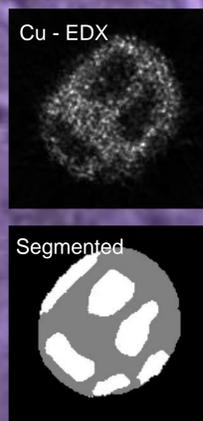


Typical EELS spectrum of CuNiFe summed over the area of a single projection

Reconstruction

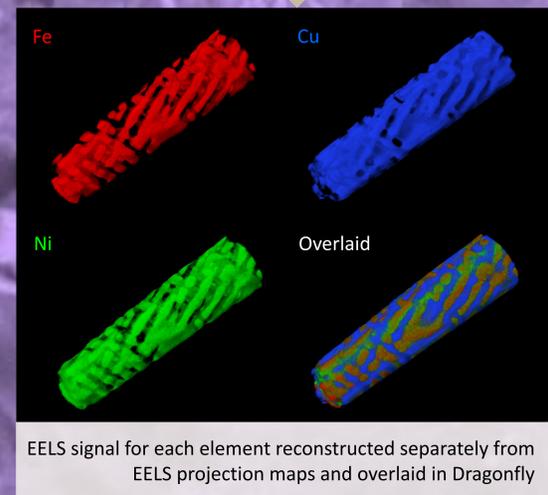
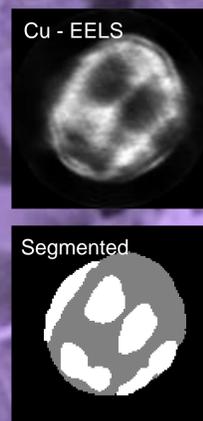


EDX signal for each element reconstructed separately from EDX projection maps and overlaid in Dragonfly



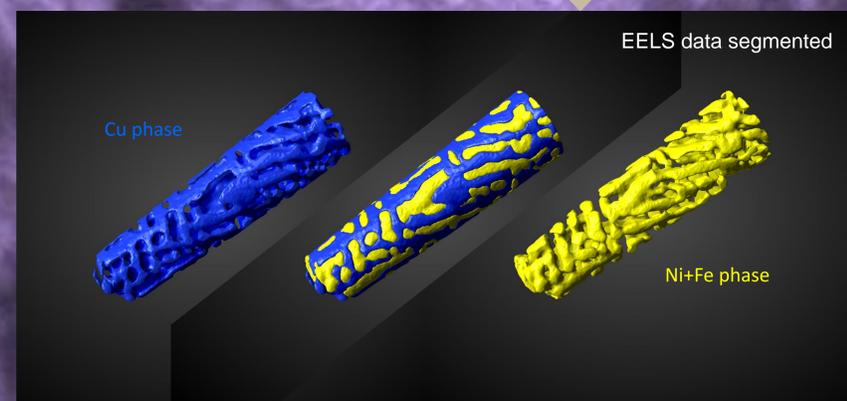
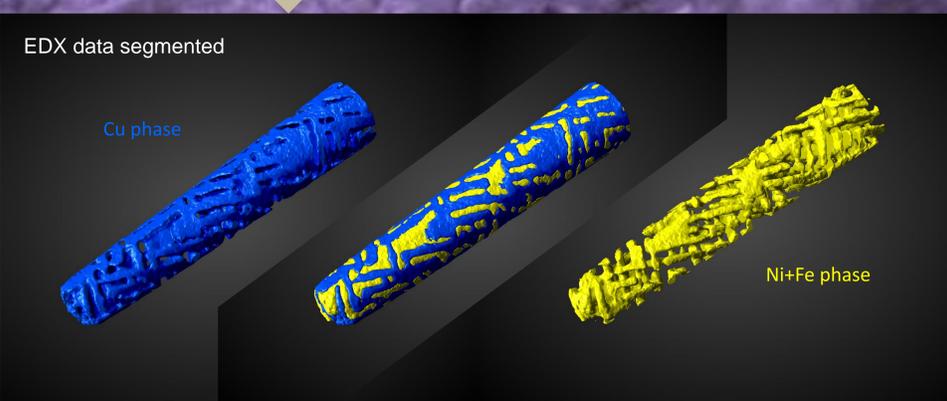
Tomography:

2 partial tilt series in 180° range with 2-5° step.
SIRT reconstruction.
Segmentation from 3 channels with Ilastik



EELS signal for each element reconstructed separately from EELS projection maps and overlaid in Dragonfly

Segmentation

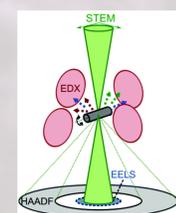


Results and discussion

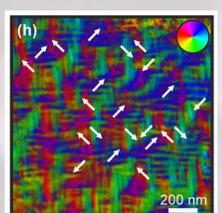
Ni+Fe phase was previously thought to be single-standing platelets alongside interconnected areas. However, our reconstruction shows that the precipitate phase is, in fact, completely interconnected, and consists of smaller flat swirl-like slab domains that are connected in a third dimension by bigger widely spaced platelets. These building blocks are anisotropically propagating in $\langle 100 \rangle$ directions. (See Figure in the Introduction)

Future questions to explore:

- A quantitative 3D model of the periodicity and domain sizes
- Presence of Ni within Cu phase
- Multimodal reconstruction using both EDX and EELS channels [1]
- Correlation with magnetic properties [2]



[1] R. Huber, et al. *Nanoscale* 11.12 (2019): 5617-5632.



[2] T. Radlinger, et al. *Journal of Alloys and Compounds* (2022): 166214.

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Contact

tatiana.kormilina@felmi-zfe.at
www.felmi-zfe.at

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