

Surface sample preparation for EPMA using GDOES

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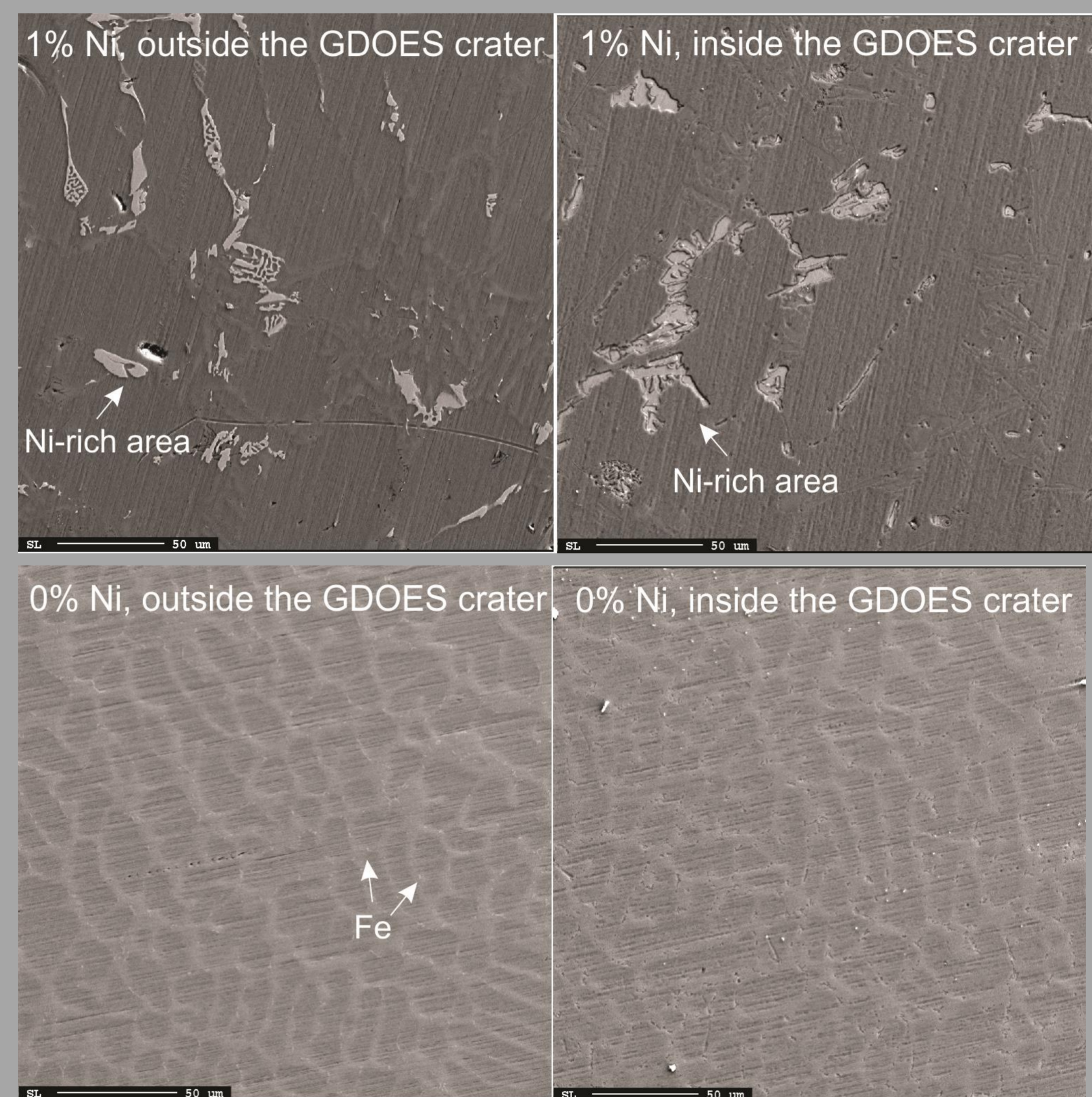


A study of 4 Al-Si cast alloy samples with varying Ni concentrations: 0, 0.5, 1, and 2 at.%. GDOES was used to measure the concentrations of the elements and Electron Probe Microanalysis (EPMA) was used to make elemental mappings and reveal the microstructure. Samples for EPMA were prepared by mechanical polishing with SiC and diamond paper down to 1 μm . GDOES was used at low power (10 W) and short time (10 s) to study the possibility of using it as a final polishing step for EPMA.

Element concentrations obtained by GDOES

	Undoped sample, %	Sample with 0.5% Ni, %	Sample with 1% Ni, %	Sample with 2% Ni, %
Al	92	92	92	91
Si	7.4	7.2	6.5	7.1
Ni	0	0.42	0.91	1.6
Fe	0.078	0.088	0.088	0.078
Mg	0.37	0.34	0.43	0.29

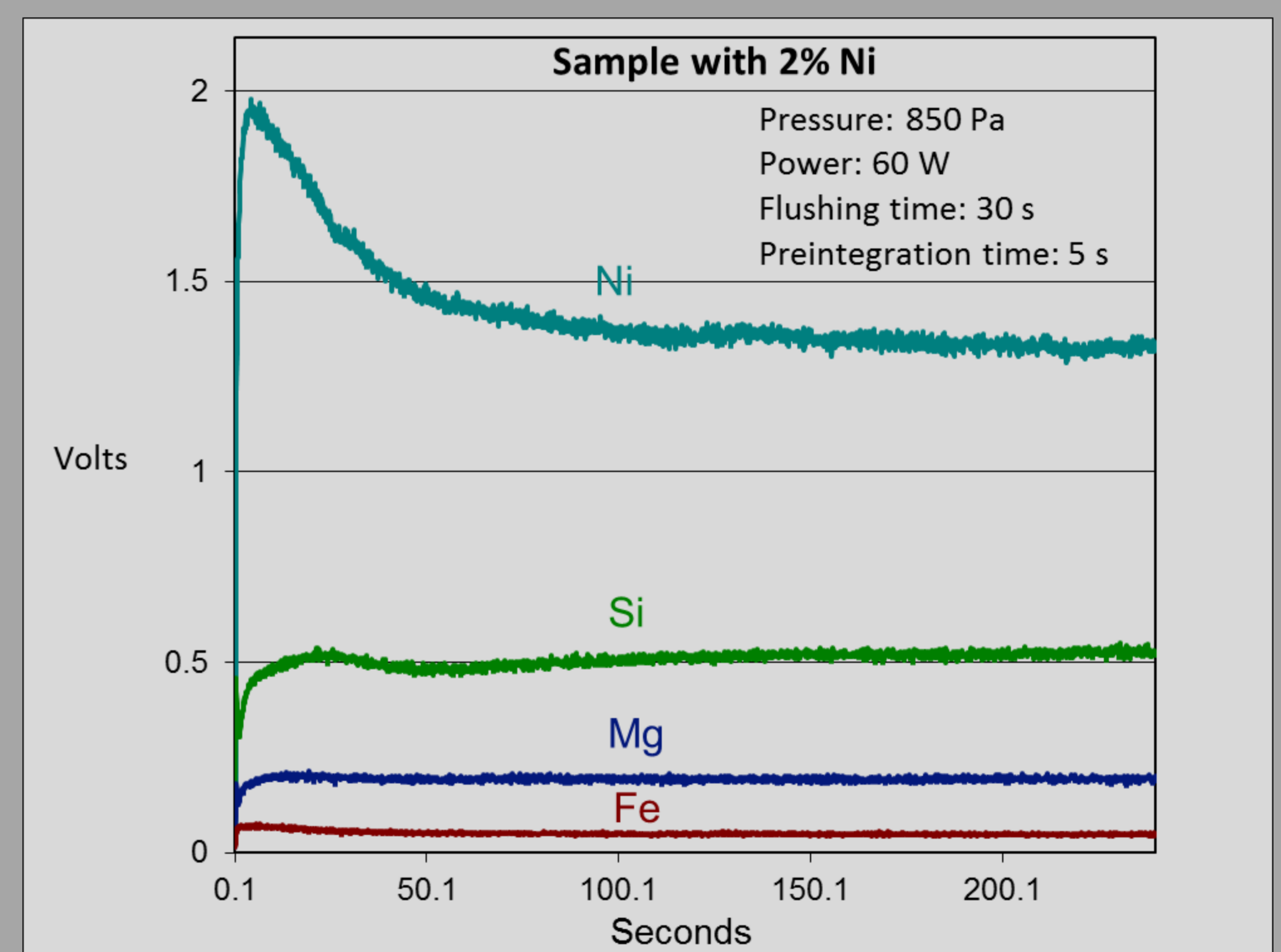
Preferential sputtering of Ni/Fe-rich areas in GDOES (polishing attempt, SEM images)



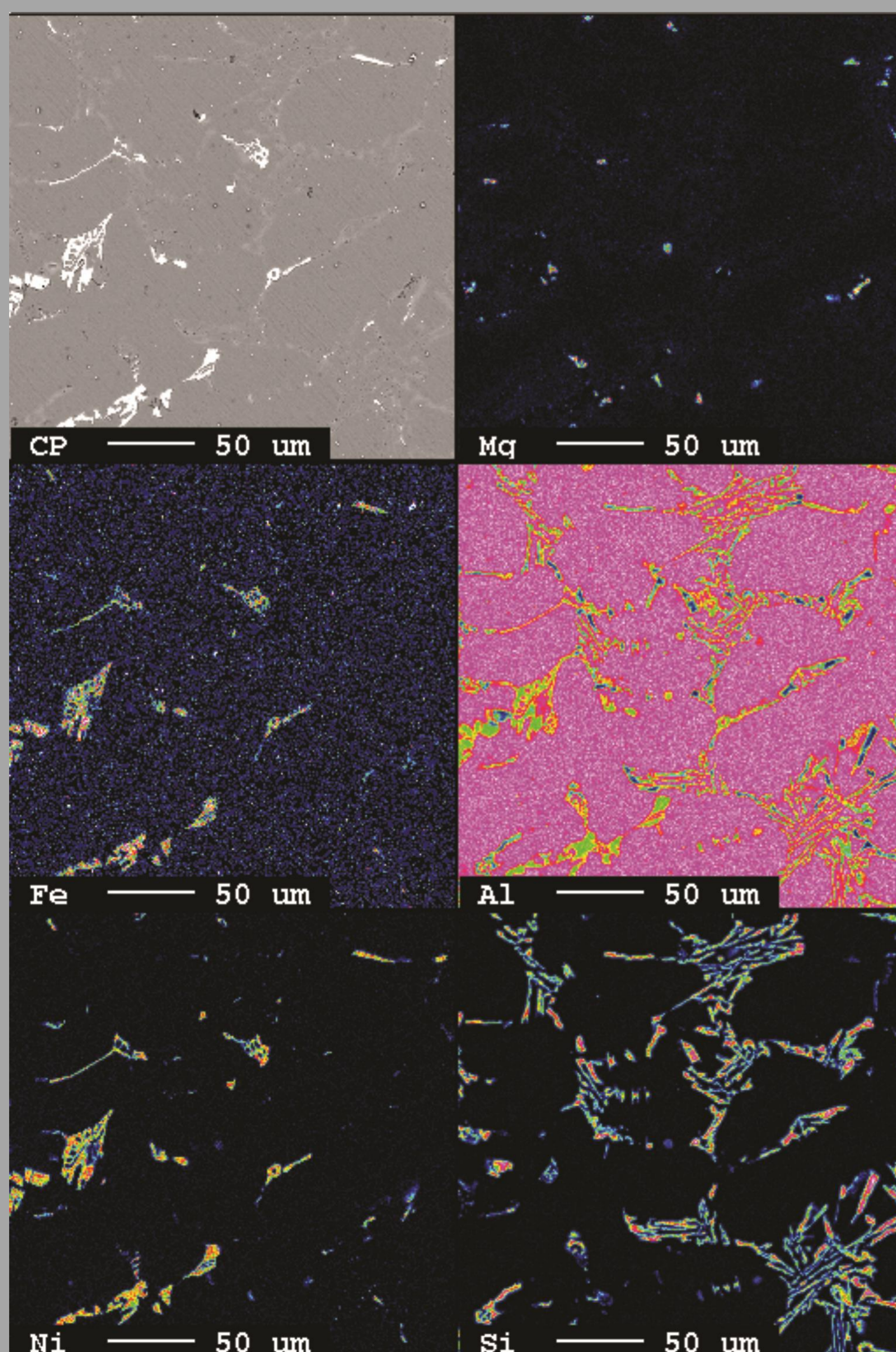
Element concentrations obtained by EDS

	Undoped sample, %	Sample with 0.5% Ni, %	Sample with 1% Ni, %	Sample with 2% Ni, %
Al	86	89	89	87
Si	7.8	10	9.8	9.8
Ni	0	0.5	0.91	1.7
Fe	0.08	0.091	0.15	0.15
Mg	0.22	0.18	0.18	0.15

GDOES depth profile



EPMA elemental maps (1% Ni sample)



Conclusions

- The results of the GDOES analysis confirm the target levels of Ni for all the 4 samples investigated
- Ni/Fe-rich areas are sticking out of the sample surface after polishing (larger hardness)
- SEM image from inside the GDOES crater (1 % N), shows Ni/Fe-rich areas preferentially etched
- Preferential etching agrees well with the relative sputtering rates for Ni (1.5), Fe (1.0) and Al (0.37)¹
- Long presputtering times (>2 min) are required for accurate bulk quantification
- GDOES as a final polishing step for EPMA or EBSD is difficult to use for these alloys due to preferential sputtering

REFERENCE

1. T. Nelis, R. Payling, Practical guide to GDOES, RSC Analytical Spectroscopy Monographs, Cambridge, UK, 2003