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Nitrogen abundance measurements by EPMA in Fe-Ni alloys: towards determining the N distribution in iron meteorites

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Quantitative determination of ultra-light elements such as nitrogen by electron probe microanalysis (EPMA), particularly in metallic matrices, remains challenging [1]. This limitation is mainly related to the low fluorescence yield of nitrogen and elemental interferences, which result in weak X-ray signals, reduced count rates, and poor signal-to-background ratios [2-3].

To measure nitrogen abundances by EPMA in Fe-Ni alloy matrices (e.g., iron meteorites and experimental samples), we synthesized reference materials by nitriding alloys with varying Ni contents (up to 10 wt.%). The main reference material (Fe_4N) was produced by nitriding pure iron, and XRD (X-ray Diffraction) analysis confirmed the presence of a single α-Fe_4N phase. $Fe_{95}Ni_5$ and $Fe_{90}Ni_{10}$ alloys were included in the nitriding process to evaluate the effect of Ni on nitrogen incorporation and potential matrix effects. For a cross-calibration, the samples were characterized using two different EPMA devices: a FEG JEOL JXA-8530F and a FEG JEOL JXA-8530F Plus equipped with a Soft X-ray Emission Spectrometer (SXES), specifically optimized for ultra-light element analysis. The characterized samples were then used as reference materials for the JEOL JXA-8230 at CRPG.

Nitrogen measurements by EPMA have been initiated on iron meteorites and on synthetic Fe-Ni alloys produced with a piston-cylinder apparatus. Five iron meteorites were selected for this study: three magmatic irons (Wiley, Walker County and Cape York) and two non-magmatic irons (Toluca and Miles). With the exception of the low-Ni Walker County meteorite, which consists of a single kamacite phase, the selected samples permit investigation of nitrogen distribution in both kamacite (α-Fe) and taenite (γ-Fe). Nitrogen distribution is also being examined in metallic phases obtained from metal-silicate partitioning experiments performed at 1 GPa and 1500 °C for 6 hours, with variable Ni proportions in the Fe-Ni alloy (0 to 20 wt.%), following [4]. Ongoing analyses focus on resolving nitrogen heterogeneity among the quenched products [e.g., 5], particularly across metallic phases showing variable Fe/Ni ratios.

References: . [1] von der Handt et al. (2019). [2] Raudsepp (1995). [3] Bastin et al. (1991). [4] Dalou et al. (2024). [5] Grewal et al. (2019).