

Supporting information

Structure and Magnetism of Iron-Substituted Nickel Hydroxide Nanosheets

Samuel W. Kimmel ^{1,‡}, Barry D. Koehne ^{1,‡}, Ben Gibson ², Wilhelmus J. Geerts ^{1,3}, Nikoleta Theodoropoulou ^{1,3} and Christopher P. Rhodes ^{1,2,*}

¹Material Science, Engineering, and Commercialization Program Texas State University, San Marcos, TX 78666, USA

²Department of Chemistry and Biochemistry, Texas State University, San Marcos, TX 78666, USA

³Department of Physics, Texas State University, San Marcos, TX 78666, USA

[‡]These authors contributed equally to this work

*corresponding authors, e-mail: cprhodes@txstate.edu; ntheo@txstate.edu

Table S1. Mass and volume of chemical precursors used for the microwave synthesis of Ni(OH)₂ and Fe-substituted Ni(OH)₂ nanosheets.

| Precursor | Ni _{1-x} Fe _x ratio | | | | |
|--|---|---------------------------------------|---------------------------------------|---------------------------------------|---------------------------------------|
| | Ni _{1.00} Fe _{0.00} | Ni _{0.95} Fe _{0.05} | Ni _{0.90} Fe _{0.10} | Ni _{0.80} Fe _{0.20} | Ni _{0.50} Fe _{0.50} |
| Ni(NO) ₂ · 6 H ₂ O (g) | 1.00 | 0.95 | 0.90 | 0.80 | 0.50 |
| FeSO ₄ · 7 H ₂ O (g) | 0.00 | 0.478 | 0.956 | 0.1434 | 0.478 |
| Urea (g) | 0.82 | | | | |
| H ₂ O (mL) | 3.0 | | | | |
| Ethylene glycol (mL) | 21.0 | | | | |

A pure solution of nickel nitrate, urea, ethylene glycol and water without any iron sulfate (Ni_{1.00}Fe_{0.00}) produces an opaque green solution before the microwave reaction. In contrast, a pure solution of iron sulfate, urea, ethylene glycol and water without any nickel nitrate (Ni_{0.00}Fe_{1.00}) produces an opaque yellow solution (Figure S1a). Substituting nickel nitrate for iron sulfate gradually changes the color of the pre-reaction solution from the characteristic green of a pure nickel nitrate solution to the yellow of a pure iron sulfate solution. After microwave heating, the colors of the reactants progressively darken from a light green (Ni_{1.00}Fe_{0.00}) to progressively darker shades of yellow (Ni_{0.95}Fe_{0.05}), orange (Ni_{0.90}Fe_{0.10}), light brown (Ni_{0.80}Fe_{0.20}), rust colored (Ni_{0.50}Fe_{0.50}) and dark brown (Ni_{0.00}Fe_{1.00}) with increasing iron content (Figure S1b); the color changes observed in the post-reaction solutions are also present during washing and centrifugation and after drying (Figure S1c-d).

a) Before microwave reaction

b) After microwave reaction

c) After washing & centrifugation

d) After drying

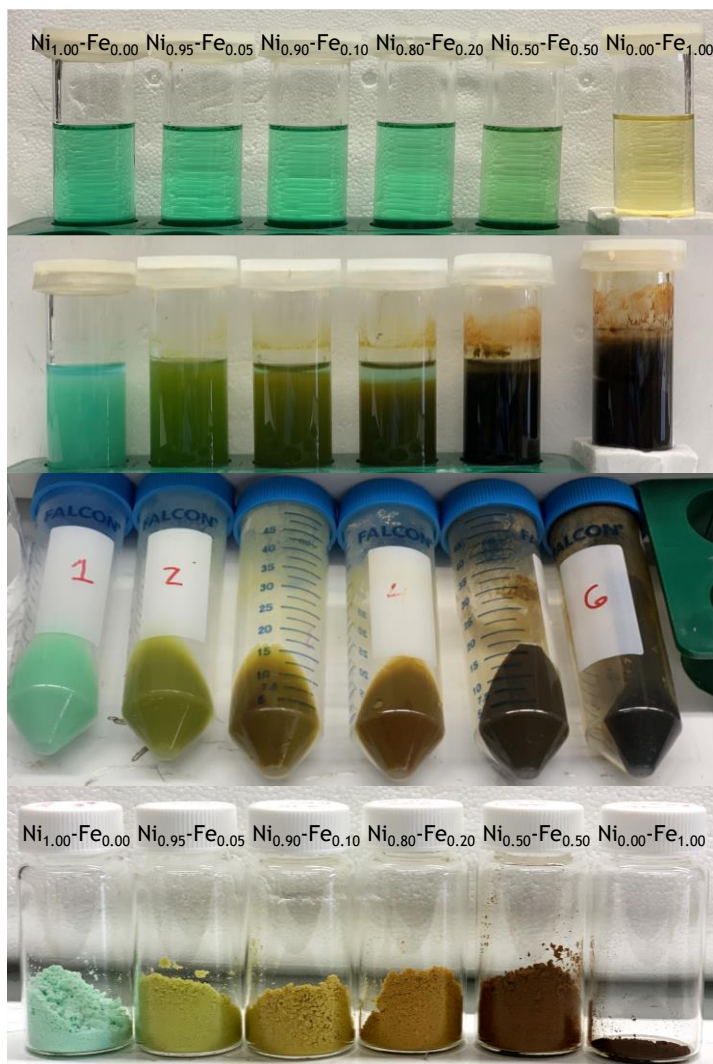


Figure S1. Photographs of the microwave synthesized iron-substituted nanosheet powders a) before microwave reaction, b) after the microwave reaction, c) after washing and centrifugation, and d) after drying.

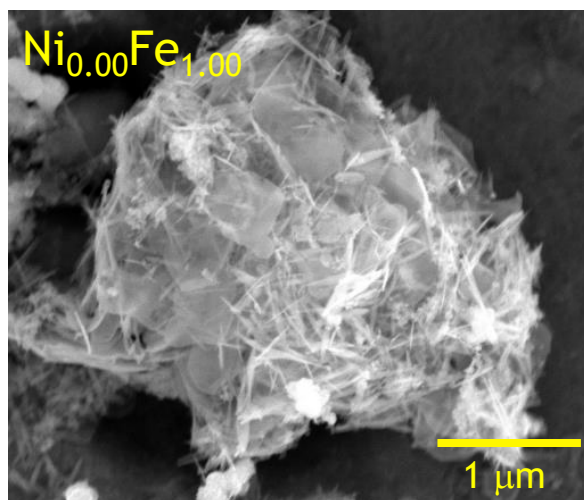


Figure S2. Scanning electron micrograph of $\text{Ni}_{0.00}\text{Fe}_{1.00}$.

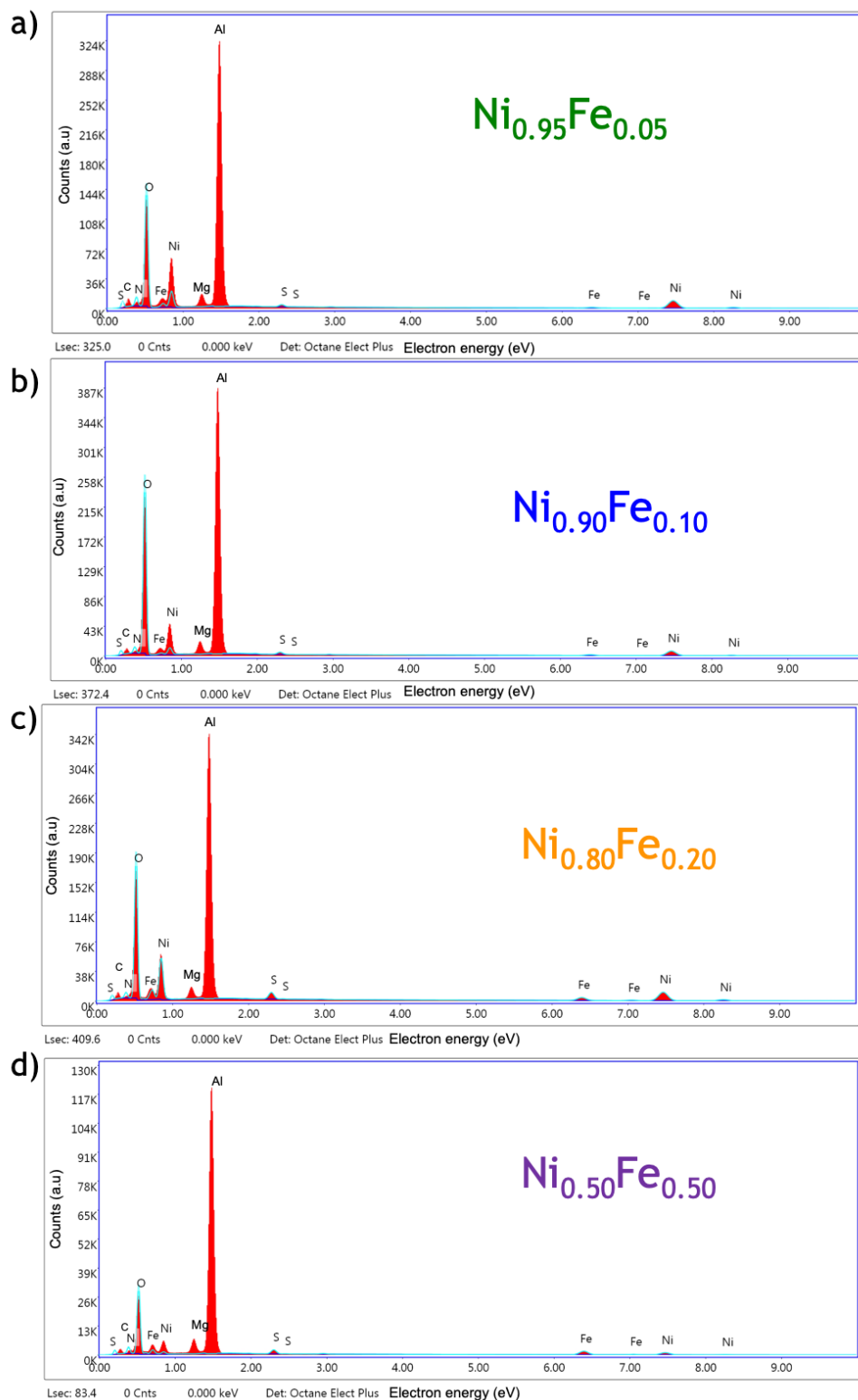


Figure S3. Energy dispersive X-ray spectra of a) $\text{Ni}_{0.95}\text{Fe}_{0.05}$, b) $\text{Ni}_{0.90}\text{Fe}_{0.10}$, c) $\text{Ni}_{0.80}\text{Fe}_{0.20}$, and d) $\text{Ni}_{0.50}\text{Fe}_{0.50}$.

Table S2. Average atomic % of elements determined by energy dispersive X-ray spectra.

| | Ni | Fe | O | N | S |
|---|--------------|-------------|--------------|-------------|-------------|
| Ni_{0.95}Fe_{0.05} | 17.87 ± 0.74 | 1.12 ± 0.11 | 66.51 ± 4.06 | 9.98 ± 0.05 | 0.91 ± 0.15 |
| Ni_{0.90}Fe_{0.10} | 12.92 ± 5.44 | 2.08 ± 1.40 | 76.78 ± 7.78 | 6.02 ± 0.84 | 1.66 ± 1.19 |
| Ni_{0.80}Fe_{0.20} | 14.46 ± 3.18 | 3.62 ± 0.13 | 73.12 ± 3.45 | 6.39 ± 0.28 | 2.41 ± 0.13 |
| Ni_{0.50}Fe_{0.50} | 8.07 ± 0.05 | 8.43 ± 0.95 | 75.85 ± 0.66 | 5.20 ± 0.47 | 2.46 ± 1.13 |

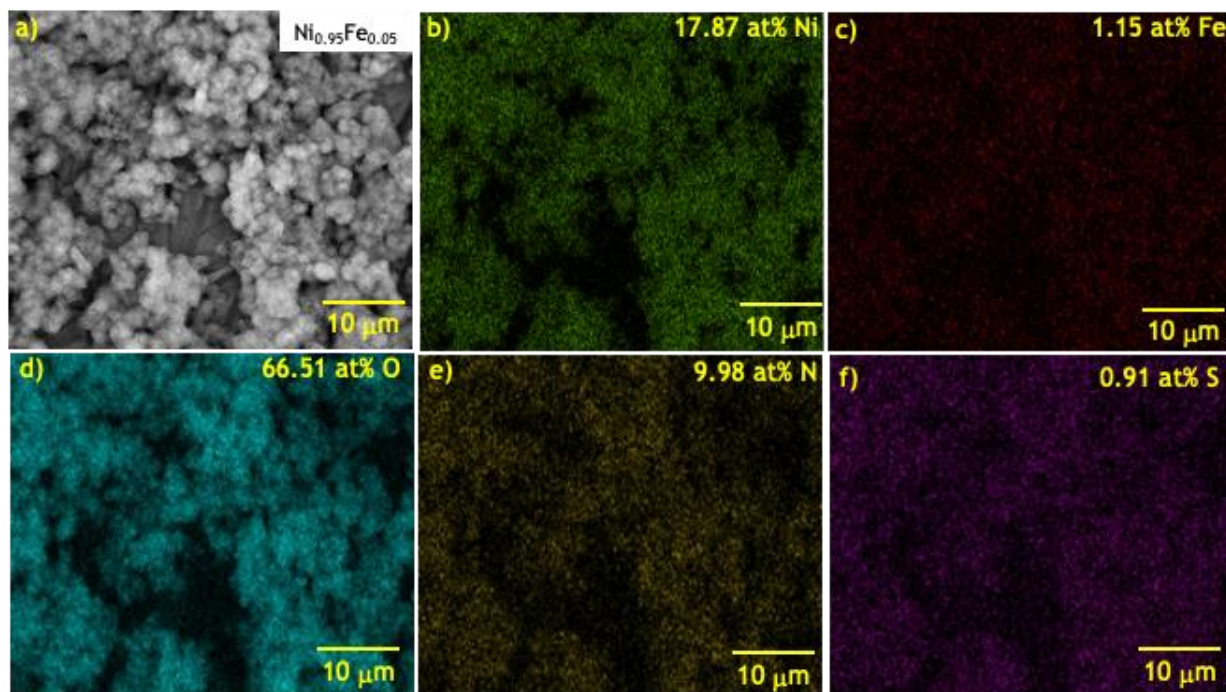


Figure S4. SEM image (a) and elemental mapping of Ni, Fe, O, N, and S (b-f) of 5% Fe-substituted Ni(OH)₂ nanosheets (Ni_{0.95}Fe_{0.05}).

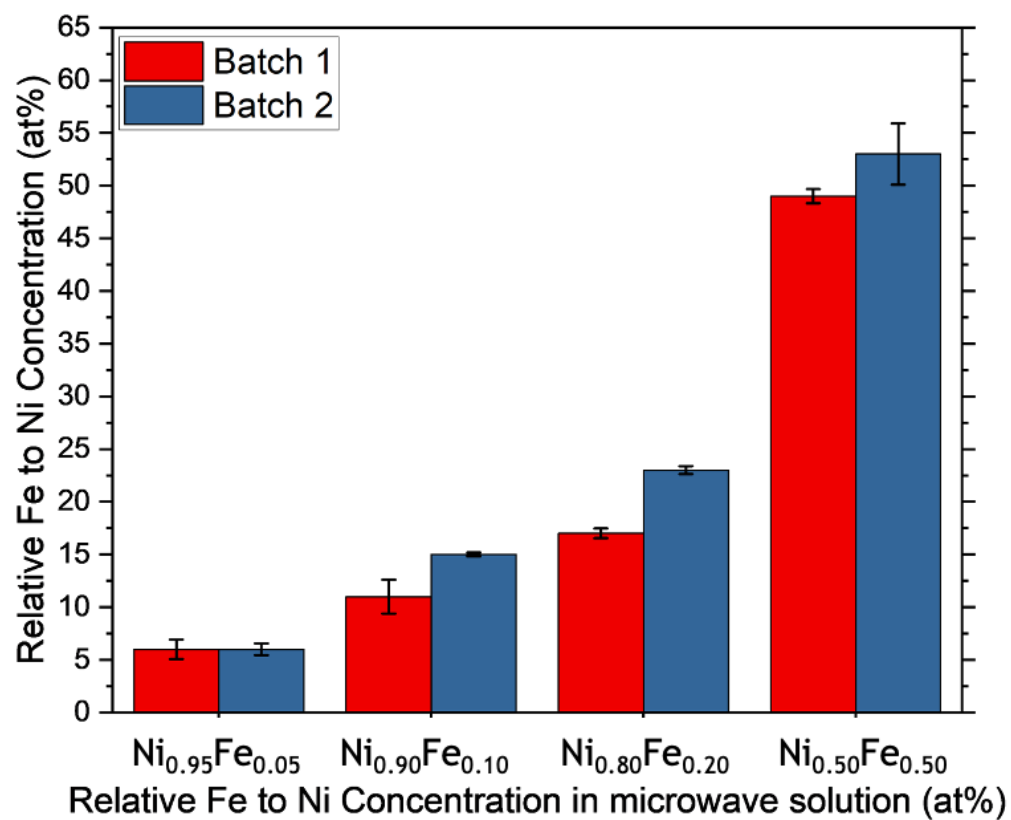


Figure S5. Comparison of the relative Fe-to-Ni concentration between two batches of material.

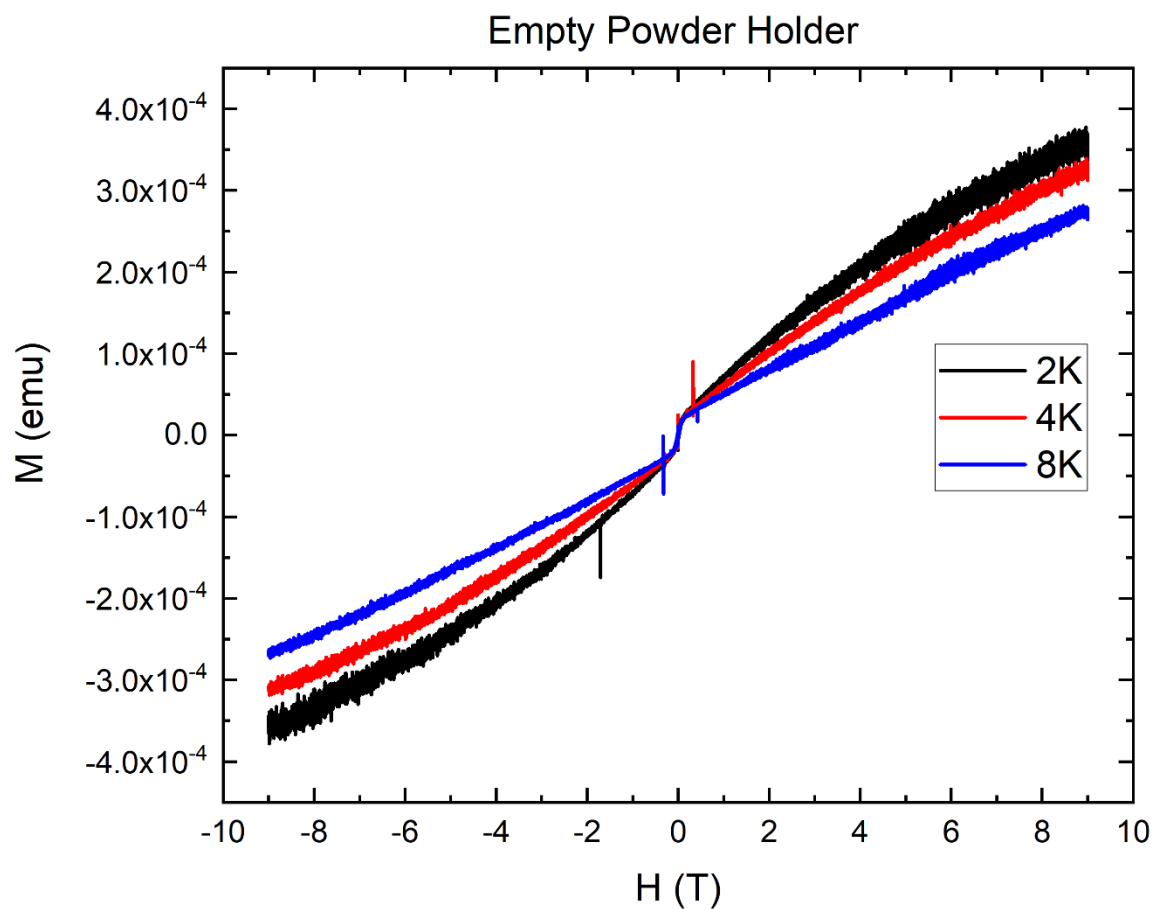


Figure S6. Magnetization versus magnetic field at 2 K, 4 K, and 8 K of an empty powder holder; the magnetization is two-three orders of magnitude smaller than the signal from the sample ensuring that the holder signal did not significantly contribute to the sample signal.

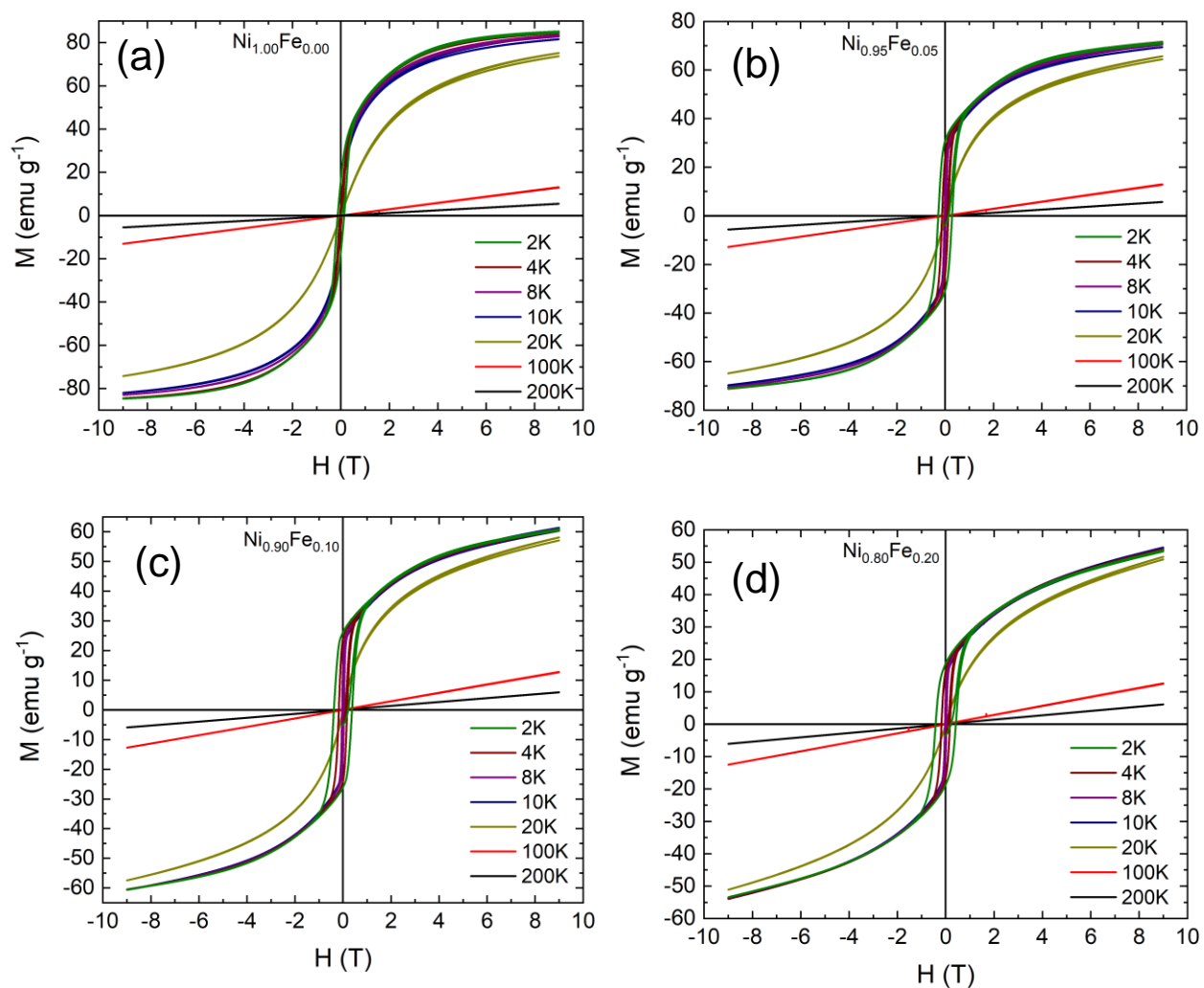


Figure S7. M vs H loops in the full range of the magnetic fields, +9 T to -9 T, at temperatures of 2-200 K for (a) $\text{Ni}_{1.00}\text{Fe}_{0.00}$ (0% Fe), (b) $\text{Ni}_{0.95}\text{Fe}_{0.05}$ (5% Fe), (c) $\text{Ni}_{0.90}\text{Fe}_{0.10}$ (10% Fe), and (d) $\text{Ni}_{0.80}\text{Fe}_{0.20}$ (20% Fe). One feature of the loops of the 10% and 20% Fe is the lack of saturation which points to the existence of noncollinear (canted) magnetic structure.

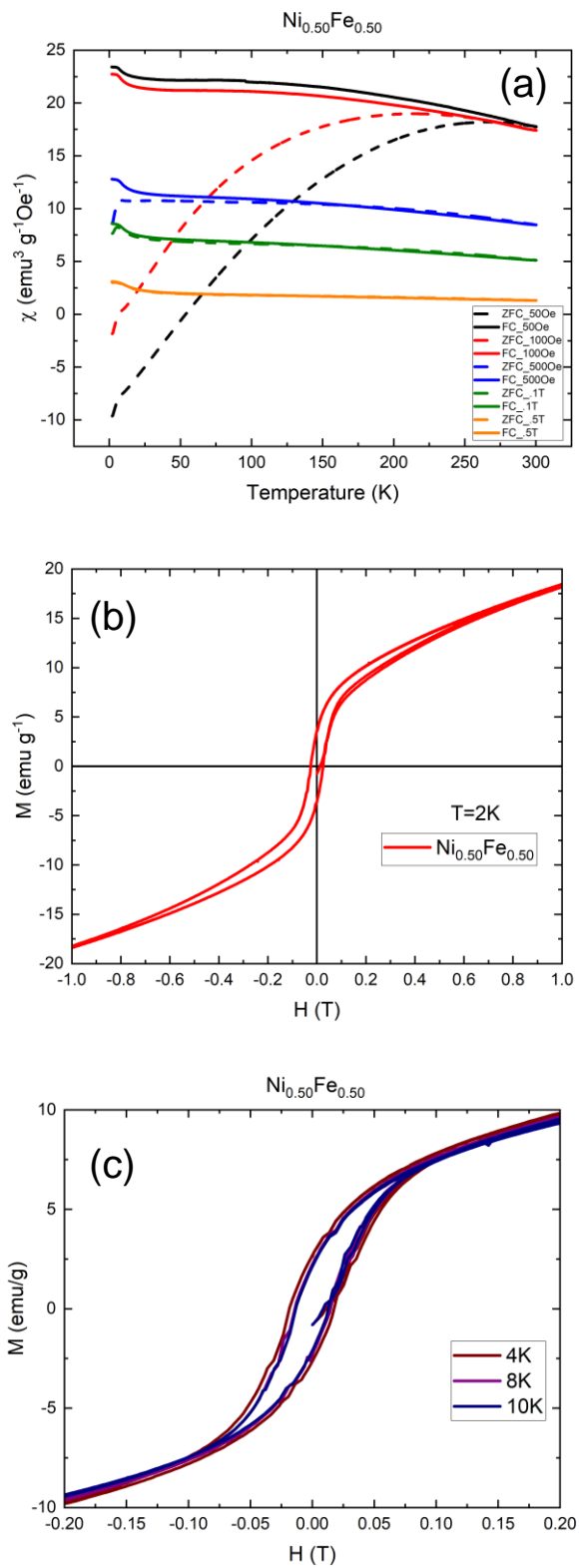


Figure S8. (a) FC and ZFC Magnetization, (b) M-H loop at 2 K, and (c) M-H loops at 4 K, 8 K, 10 K for 50 % Fe ($\text{Ni}_{0.50}\text{Fe}_{0.50}$).