Supplementary Information

§1. Rietveld analysis and electron diffraction

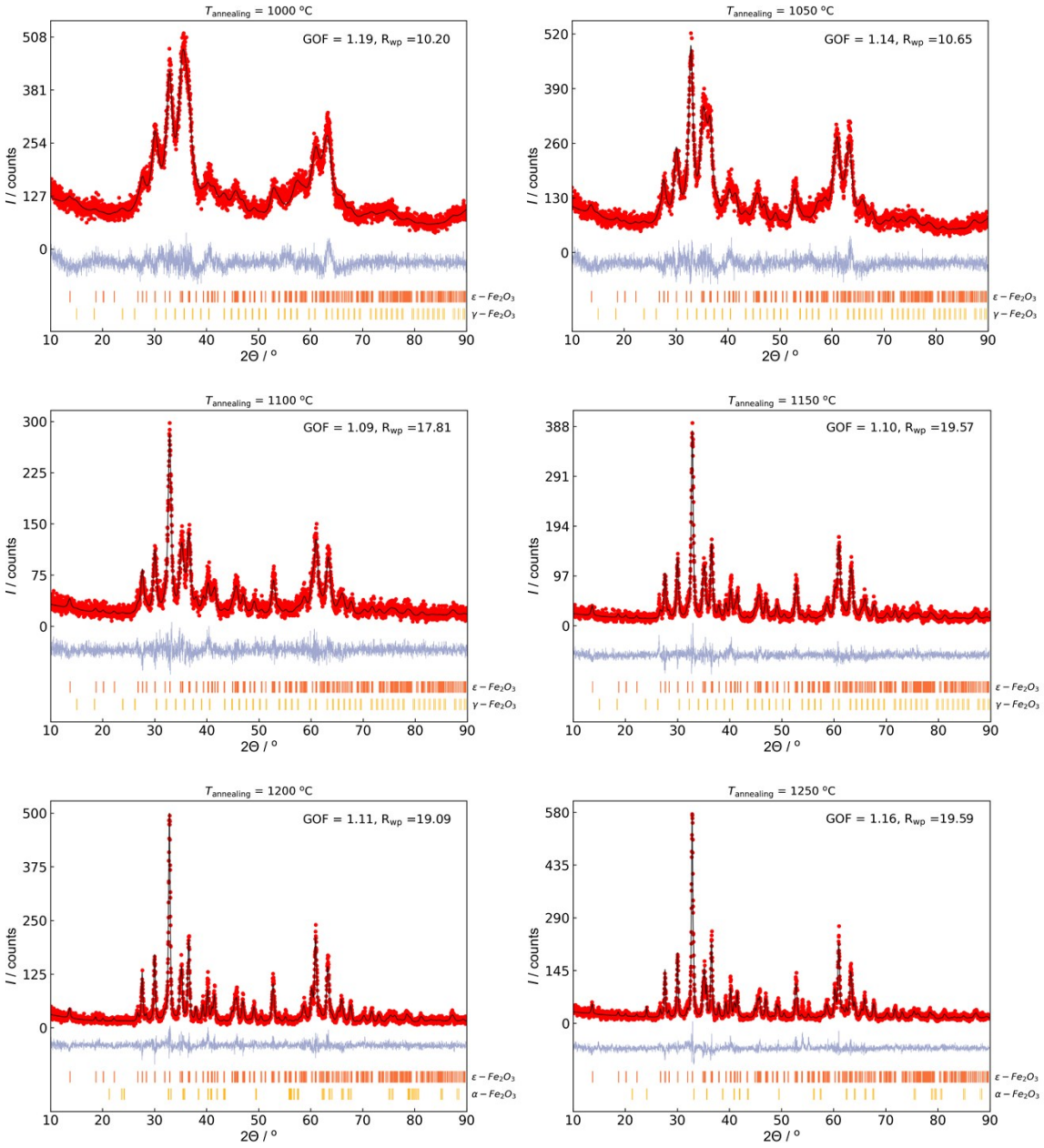
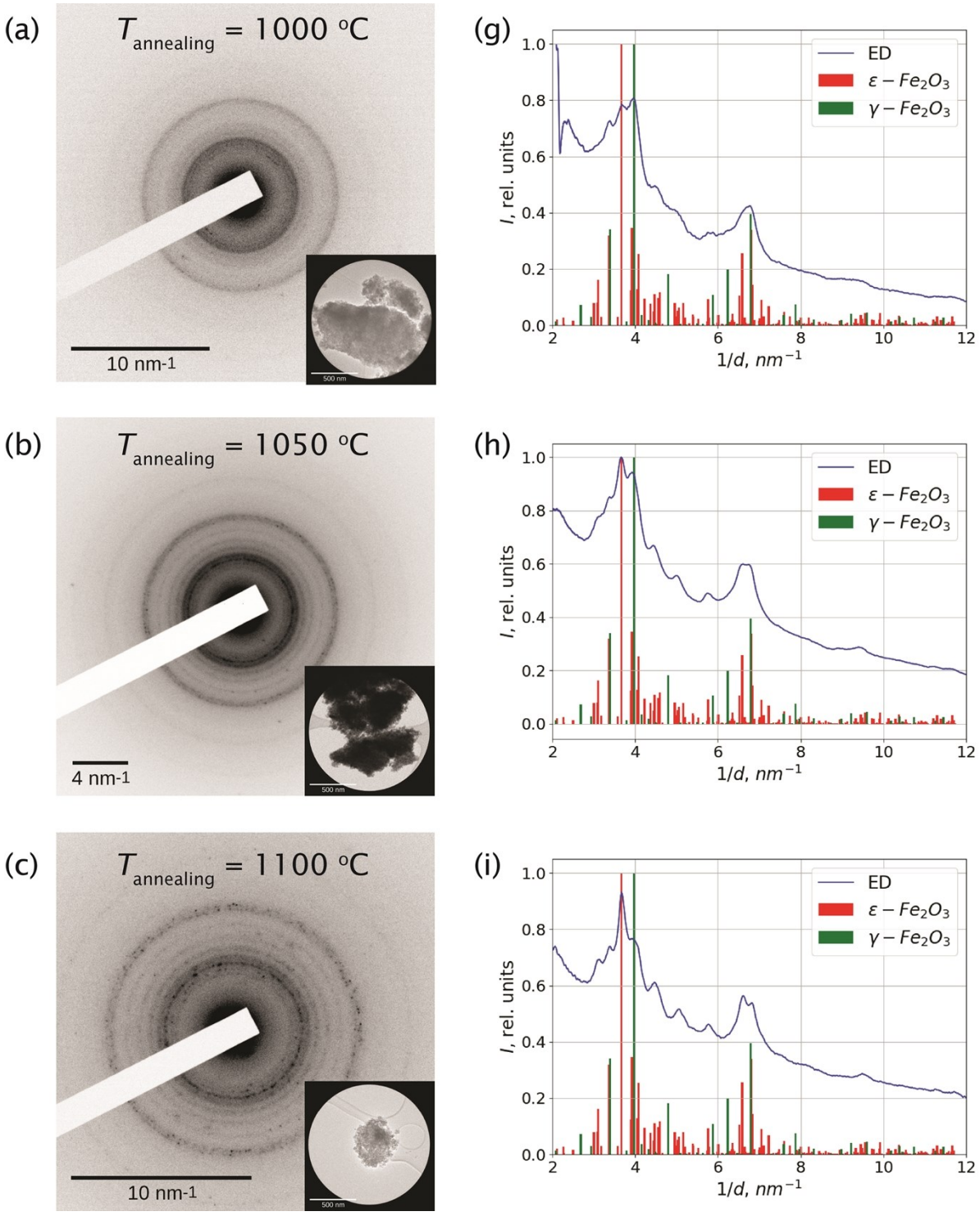


Figure S1. Full-profile analysis of the washed iron oxide samples obtained at various temperatures.


(a)
$T_{annealing}$ = 1000 °C
10 nm-1
500 nm
(b)
$T_{annealing}$ = 1050 °C
4 nm-1
500 nm
(c)
$T_{annealing}$ = 1100 °C
10 nm-1
500 nm
(g)
(h)
(i)
ED
$\varepsilon - Fe_2O_3$
$\gamma - Fe_2O_3$
I, rel. units
1/d, nm^{-1}

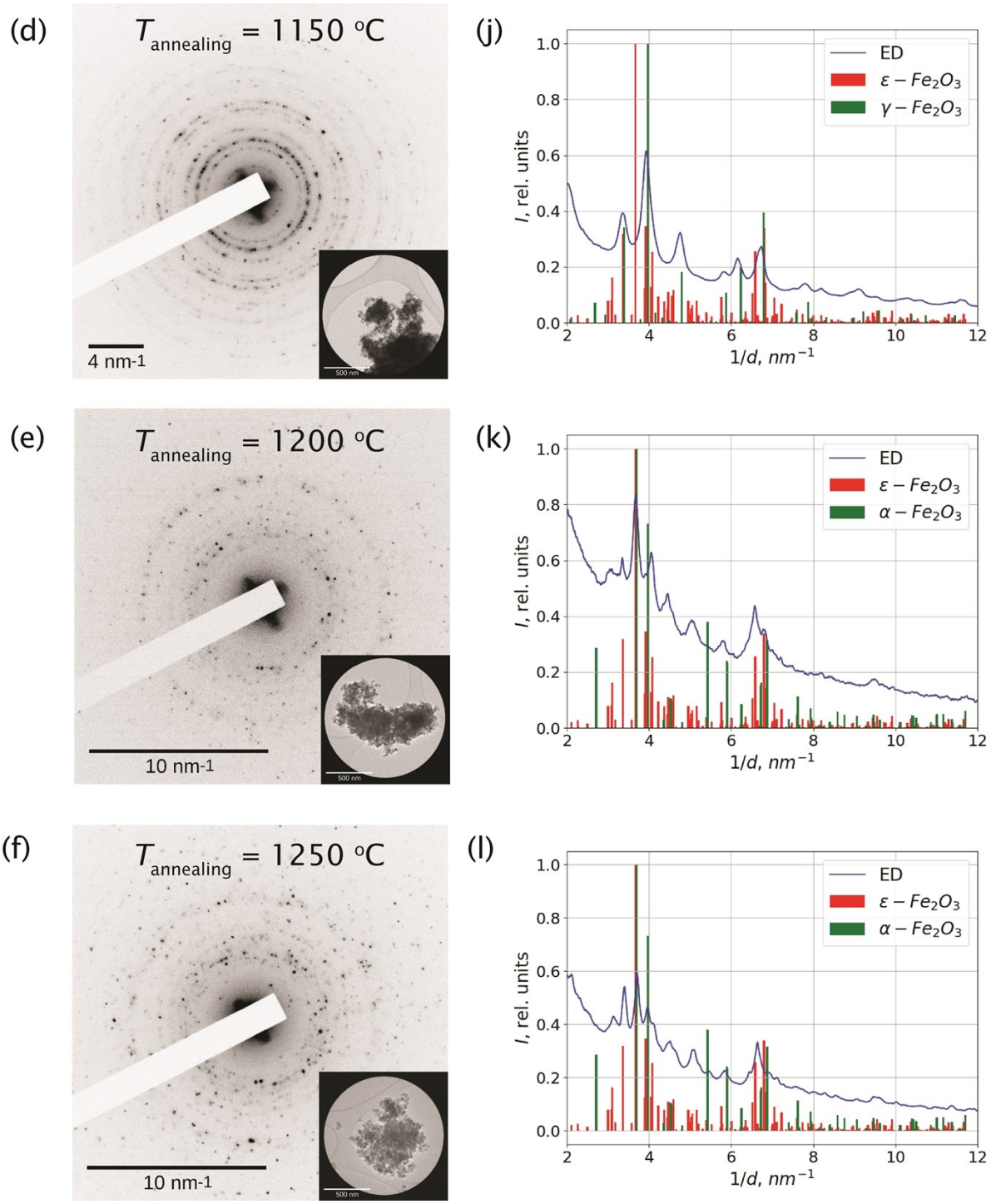


Figure S2. Electron diffraction patterns (a – f) and radial-integrated ones (g – l) of the washed iron oxide samples obtained at various temperatures.

§2. FMR frequency modeling

According to the theory from ref. [I. S. Poperechny et al, *Phys. Rev. B*, 93(1), 014441, 2016] the frequency of ferromagnetic resonance for superparamagnetic particle can be expressed as:

$$Q_r = 1 - 2\varepsilon\frac{L_2(\xi_L)}{L_1(\xi_L)}P_2(\cos\psi),$$

where P_2 is the second Legendre polynomial, $L_1(x) = 1/\tanh x - 1/x$ and $L_2(x) = 1 - 3L_1(x)/x$ are Langevin functions, ψ – easy axis tilt angle, $\varepsilon = \frac{\gamma H_a}{2\omega} = \frac{\gamma H_a}{2\gamma H_a} = 1/2$, $\xi_L = \frac{\mu\omega}{\gamma k_B T} = \frac{M_S V \gamma K_u}{M_S \gamma k_B T} = \frac{K_u V}{k_B T}$, where ω – FMR frequency, H_a – anisotropy field, γ – gyromagnetic ratio, T – temperature, k_B – Boltzmann constant, K_u – magnetic anisotropy constant, V – particle volume.

The resonance frequency for ensemble of randomly oriented magnetic particles we have determined as:

$$f_r = 0.5\cdot(f_r(\psi = \pi/2) + f_r(\psi = 0)) = \frac{L_2\left(\frac{K_u V}{k_B T}\right)}{2L_1\left(\frac{K_u V}{k_B T}\right)}(P_2(\cos(\psi = 0)) +$$

,

and the value, that is proportional to the linewidth as:

$$\Delta f_r = f_r(\psi = \pi/2) - f_r(\psi = 0) = \frac{L_2\left(\frac{K_u V}{k_B T}\right)}{L_1\left(\frac{K_u V}{k_B T}\right)}(P_2(\cos(\psi = 0)) - P_2(\cos(\psi = \pi/2))).$$

To calculate the f_r and the Δf_r for an ensemble of randomly oriented ε-Fe_2O_3 particles the following parameters T = 300 K, K_u = 7.7·10^6 erg/cm^3, M_S = 15 emu/g, d = 0 – 50 nm^3, k_B = 1.380649·10^{-23} J/K were taken.

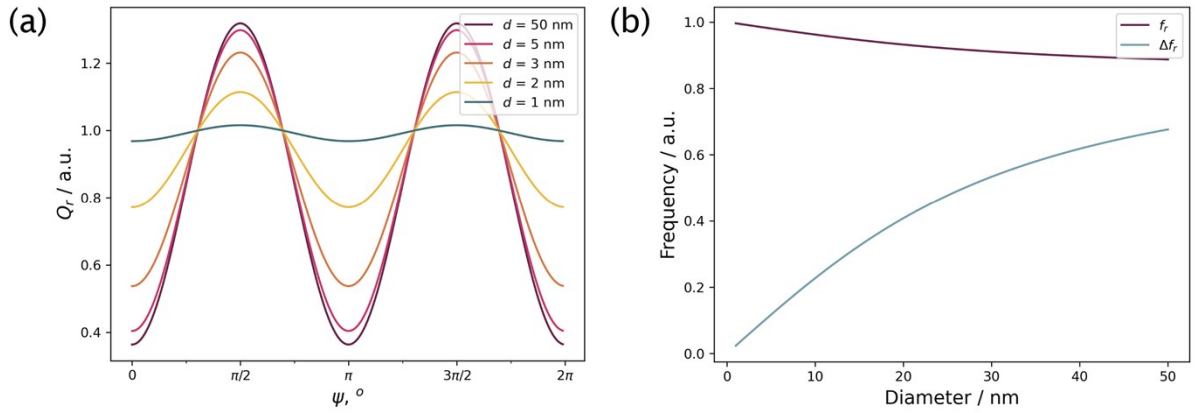


Figure S3. a) Q_r versus ψ for an epsilon oxide particle of various diameters; b) Ferromagnetic resonance frequency f_r and linewidth Δf_r ($\gamma \sim \Delta f_r$) as functions of the particle diameters in an ensemble of randomly oriented spherical ε-Fe_2O_3 particles with parameters: K_u = 7.7·10^6

erg/cm^3 [18], M_S = 15 emu/g. Data was calculated using the theory from ref. [I. S. Poperechny et al, *Phys. Rev. B*, 93(1), 014441, 2016].