## Supplementary Information

To confirm the presence of the  $\varepsilon$ -Fe<sub>2</sub>O<sub>3</sub> phase, we studied the nanocomposite ( $\rho r = 0.15$ ) treated at 1000 °C by Mössbauer spectroscopy at room temperature. The Mössbauer spectrum was recorded using a standard transmission geometry equipped with a conventional constant acceleration spectrometer and a <sup>57</sup>Co source diffused into a Rh matrix. The hyperfine parameters were refined by using MOSFIT, a lorentzian linefitting program.[S1]



Figure S1 : Mössbauer spectrum obtained at 300 K of the nanocomposite  $\rho$ r=0.15 treated at 1000 °C recorded at room temperature.

The hyperfine structure can be well described by the presence of five sextets. The refinement parameters with these five components are shown in Table S1. The sextet with the high hyperfine field is assigned unambiguously to hematite. The other four components are characteristic of  $\epsilon$ -Fe<sub>2</sub>O<sub>3</sub> phase. Their proportions are quite similar, indicating thus the presence of four sites with equal occupation rate, assuming that all of iron atoms in  $\epsilon$ -Fe<sub>2</sub>O<sub>3</sub> have the same fraction without recoil. The isomer shift values ( $\delta$ ) indicate the existence of three octahedral and one tetrahedral site. Two octahedral sites are characterized by similar values of the hyperfine field, B<sub>hyp</sub> ( $\approx$  44 T), and different values of the quadrupole displacement 2 $\epsilon$ . The third octahedral site has a smaller hyperfine field ( $\approx$  40 T). These values of B<sub>hyp</sub> are lower than those of the hematite and maghemite ( $\approx$ 50 T).

Componants	%		$\delta$ (mm.s <sup>-1</sup> )		$2\varepsilon (\text{mm.s}^{-1})$		<b>B</b> <sub>hyp</sub> ( <b>T</b> )	
	Our	Tronc	Our	Tronc	Our	Tronc	Our	Tronc
	study	et al.	study	et al.	study	et al.	study	et al.
Sextet 1 (a)	$32 \pm 2$	29	0.36	0.37	-0.11	-0.11	52 ± 1	51.5
Sextet 2 (ɛ)	16 ± 2	17	0.39	0.37	-0.19	-0.19	44 ± 2	45
Sextet 3 ( $\epsilon$ )	16 ± 2	17	0.38	0.39	-0.06	-0.06	46 ± 2	45.2
Sextet 4 ( $\epsilon$ )	17 ± 2	19	0.38	0.38	0	0	<i>40</i> ± <i>2</i>	39.5
Sextet 5 ( $\epsilon$ )	18 ± 2	18	0.21	0.21	-0.07	-0.07	26 ± 2	26.2

 Table S1: Refinement parameters obtained with five components of the Mössbauer spectrum, compared to those obtained by Tronc et al. for a similar sample [S2].

Figure S2 : Refinements of the diffraction patterns of three nanocomposites annealed at 1400 °C :  $\rho_r$  = 0.04,  $\rho_r$  = 0.15 and  $\rho_r$  = 0.95.





Sample  $\rho_r = 0.15$ 



Sample p<sub>r</sub>=0.95



	Phase	A,b,c	<d> Rms (10<sup>-4</sup>)</d>		%	R <sub>exp</sub>	R <sub>w</sub>	χ <sub>i</sub> <sup>2</sup>
		(nm)	(nm)	Microcontrain				
ρ <sub>r</sub> = 0.15	α	a=0.527 (±0.002)	113	8.8 (±0.5)	8.5	11.86	9.09	1.70
		b=1.372 (±0.002)	(±5)					
	3	a=0.510 (±0.002)	55 (±2)	6.61 (±0.5)	4.7			
		b=0.878 (±0.002)						
		C=0.946 (±0.002)						
	SiO <sub>2</sub>	a=0.496 (±0.002)	115	2.72 (±0.5)	86.8			
		b=0.694 (±0.002)	(±2)					
		0.500 (+0.000)						
$\rho_{\rm r} = 0.04$	α	a=0.529 (±0.002)	91 (±5)	7.65 (±0.5)	2.3	11.78	9.68	1.48
		D=1.372 (±0.002)						
	3	a=0.510 (±0.002)	45 (±2)	9.25 (±0.5)	2.6			
		b=0.877 (±0.002)						
		c=0.945 (±0.002)						
	SiO <sub>2</sub>	a=0.496 (±0.002)	163	2.77 (±0.5)	95.1			
		b=0.693 (±0.002)	(±5)					
ρ <sub>r</sub> = 0.95	α	a=0.528 (±0.002)	123	7.3 (±0.5)	95.3	10.73	7.94	1.82
		b=1.374 (±0.002)	(±10)					
	SiO <sub>2</sub>	a=0.496 (±0.002)	165	2.71 (±0.5)	4.7			
		b=0.694 (±0.002)	(±5)					

Table S2 : Refined values of lattice parameter of the initial  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub> composites annealed at 1400 °C for different mass fraction  $\rho_r$  = 0.04, 0.15, 0.95.

## **Complete References**

[S1] J. Teillet, F. Varret, Unpublished MOSFIT program.[S2] E. Tronc, C. Chanéac and J. P Jolivet, Journal of Solid State Chemistry 1998, 139, 93-104