Chemical Vapor Synthesis of Iron and Iron Oxide Nanoparticles

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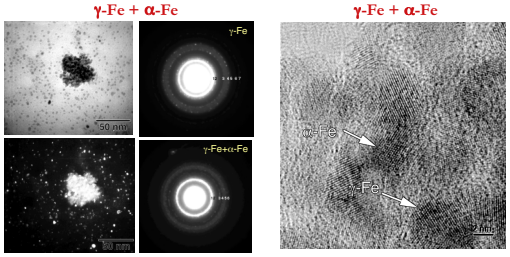
Scientific Achievement

- Iron and iron oxide nanoparticles were synthesized by chemical vapor decomposition of n-butylferrocene precursor gas in a hot-walled deposition system.
- The effects of variations in reactor chamber pressure, temperature, precursor flow-rate, and oxygen:nitrogen supply gas ratio on the structure, composition, size and size distribution of the particles were studied.
- The results indicate that nanoparticles with diameters ranging from 3 to 40 nm can be produced controllably and with a reasonably narrow size distribution.
- Oxygen content in the flow gas has the most dominant effect on the structure, composition, and size of the particles.
- Without oxygen, γ-iron and α-iron nanoparticles with diameters of 2–8 nm were observed. A small amount of oxygen in the flow gas led to the formation of larger fcc Fe_3O_4 (magnetite, Fd3m, a = 8.20 A) nanoparticles, together with core-shell structures consisting of a γ-iron metallic core surrounded by a shell of iron oxide.
- With increasing oxygen flow, the nanoparticles were observed to increase in size. Concomitantly, for larger particles the metallic core was found to exhibit the bcc α-iron structure.
- With further increase in oxygen in the flow gas, coarse particles of b-Fe_2O_3 (bcc, Ia3, a = 9.27Å) were observed, together with fine particles of Fe_3O_4. Finally, at very high oxygen partial pressure, the β-Fe_2O_3 was replaced by coarse α-Fe_2O_3 (Hematite, R3-c, a = 5.03Å, b = 13.73Å c = 2.73Å) particles.

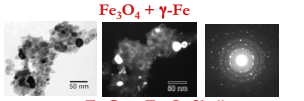
800°C, 0% Oxygen, 50 Torr

γ-Fe + α-Fe

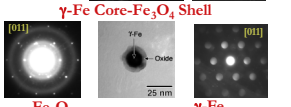


800°C, 10% Oxygen, 50 torr

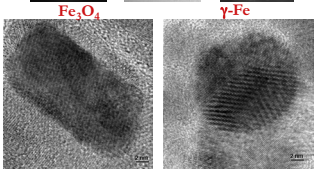
Fe_3O_4 + γ-Fe



γ-Fe Core-Fe_3O_4 Shell



Fe_3O_4 γ-Fe

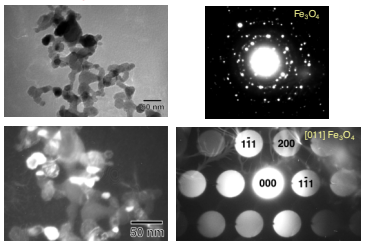


Typical Analysis of Fe_3O_4 Ring Pattern

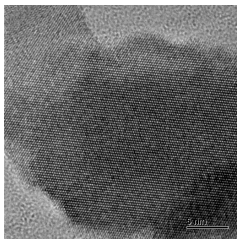
Ring No.	hkl	d (Å)
1	111	4.73
2	220	2.90
3	311	2.47
4	400	2.05
5	422	1.67
6	511	1.58
7	440	1.45
8	444	1.18
9	642	1.10
10	731	1.07
11	844	0.84

800°C, 5% Oxygen, 50 torr

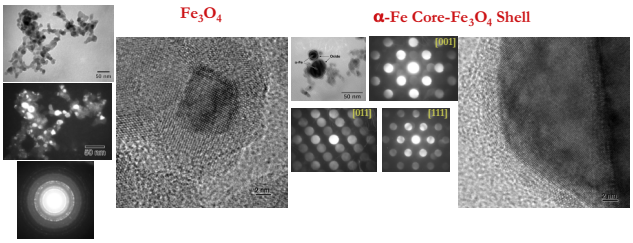
Fe_3O_4



Fe_3O_4

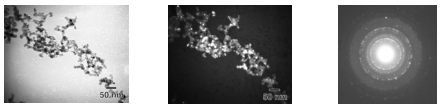


800°C, 20% Oxygen, 50 torr

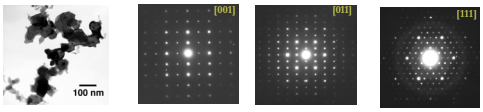


800°C, 40% Oxygen, 50 torr

Fe_3O_4

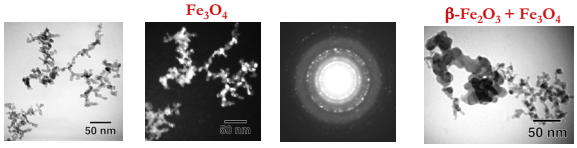


β-Fe_2O_3

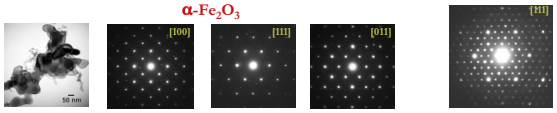


800°C, 60% Oxygen, 50 torr

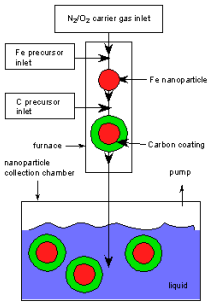
Fe_3O_4 β-Fe_2O_3 + Fe_3O_4



α-Fe_2O_3



Synthesis of Nanoparticles



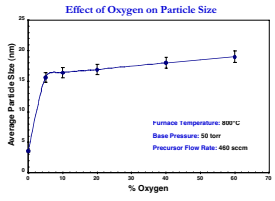
Variables Studied

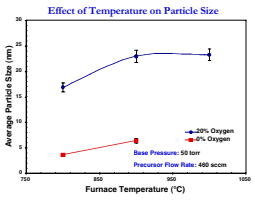
- Furnace Temperature
- Oxygen:Nitrogen Ratio
- Base Pressure
- Precursor Flow Rate
- Bubbler Temperature
- Time

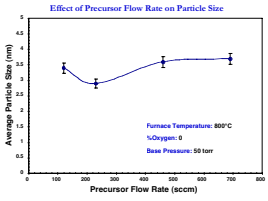
Synthesis Conditions, Structure and Size of Nanoparticles

Sample Id	Furnace Temperature (°C)	Base Pressure (Torr)	Precursor Flow Rate (sccm)	O2:N2 Ratio %	Phases	Lattice Parameters (Å)	Average Particle Size (nm)
CVC-4	1000	50	460	20	Fe_3O_4	8.20	23.3
					α-Fe	2.89	
CVC-5	900	50	460	20	Fe_3O_4	8.23	23.0
					α-Fe	2.95	
CVC-8	900	50	460	0	γ- Fe	3.54	6.4
					α-Fe	2.83	
CVC-13	800	100	460	0	γ- Fe	3.54	3.6
					α-Fe	2.83	
CVC-14	800	50	460	0	γ- Fe	3.52	5.2
					α-Fe	2.86	
CVC-15	800	200	460	0	γ- Fe	3.53	3.9
					α-Fe	2.87	
CVC-16	800	25	460	0	γ- Fe	3.53	3.4
					α-Fe	2.88	
CVC-17	800	50	690	0	γ- Fe	3.54	3.7
					α-Fe	2.83	
CVC-18	800	50	230	0	α-Fe	2.86	2.9
CVC-19	800	50	120	0	α-Fe	2.86	3.4
					Fe_3O_4	8.20	
CVC-20	800	50	460	10	γ- Fe	3.53	16.4
					α- Fe	2.84	
					Fe_3O_4	8.18	
CVC-21	800	50	460	5	γ- Fe	3.55	15.6
					α- Fe	2.87	
CVC-22	800	50	460	20	Fe_3O_4	8.19	16.9
					α- Fe	2.85	
CVC-24	800	50	460	40	Fe_3O_4	8.19	18.0
					β-Fe_2O_3	9.27	
					α-Fe	2.86	
CVC-25	800	50	460	60	Fe_3O_4	8.20	19.0
					β-Fe_2O_3	9.27	
					α-Fe_2O_3	5.03 13.73 2.73	

α-Fe - BCC γ-Fe - FCC Fe_3O_4-Fd3m β-Fe_2O_3-Ia3 α-Fe_2O_3-R3-c







Acknowledgement

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