Lecture 10

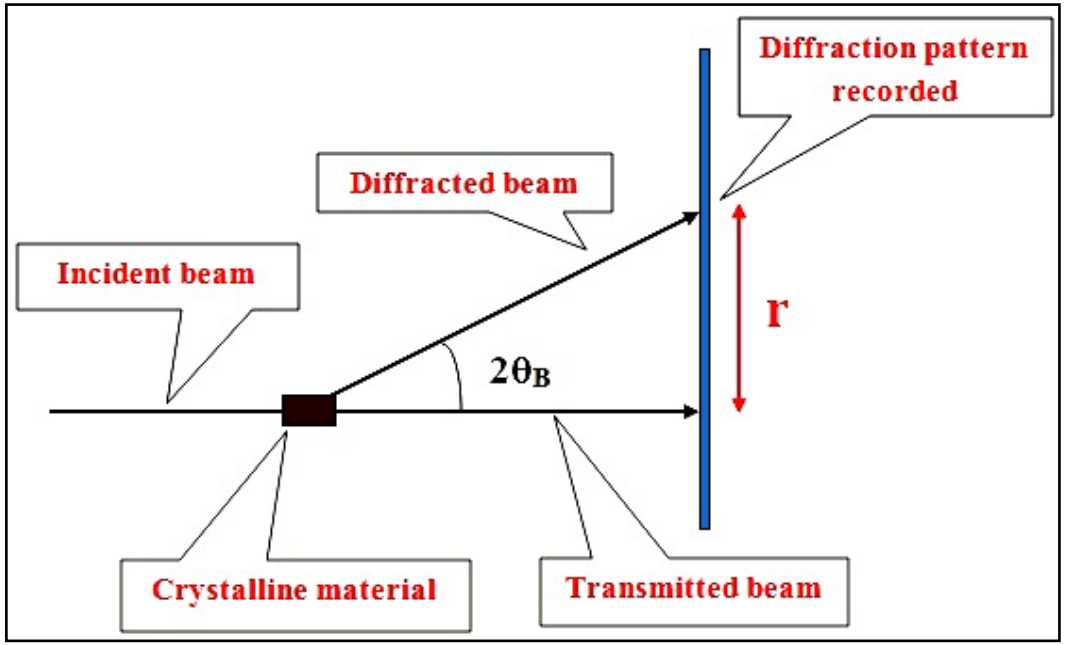
Characterization Instruments

Some basic Nanoparticles characterization techniques

- X- ray diffraction technique
- Spectroscopy techniques
  - X- ray photoelectron Spectroscopy (XPS)
  - Vibrational (Fourier transform infrared) spectroscopy (FTIR)
  - Energy Dispersive X-ray spectroscopy (EDX)
- Microscopy techniques
  - Transmission electron microscope (TEM)
  - Field emission scanning electron microscope (FESEM)
- Thermal analysis
  - Thermogravimetric Analysis (TGA)
  - Differential thermal analysis (DTA)
  - Differential scanning calorimetry (DSC)
- Brunauer-Emmet-Teller (BET) surface area technique
- Vibrating sample magnetometer (VSM)

X- ray diffraction technique

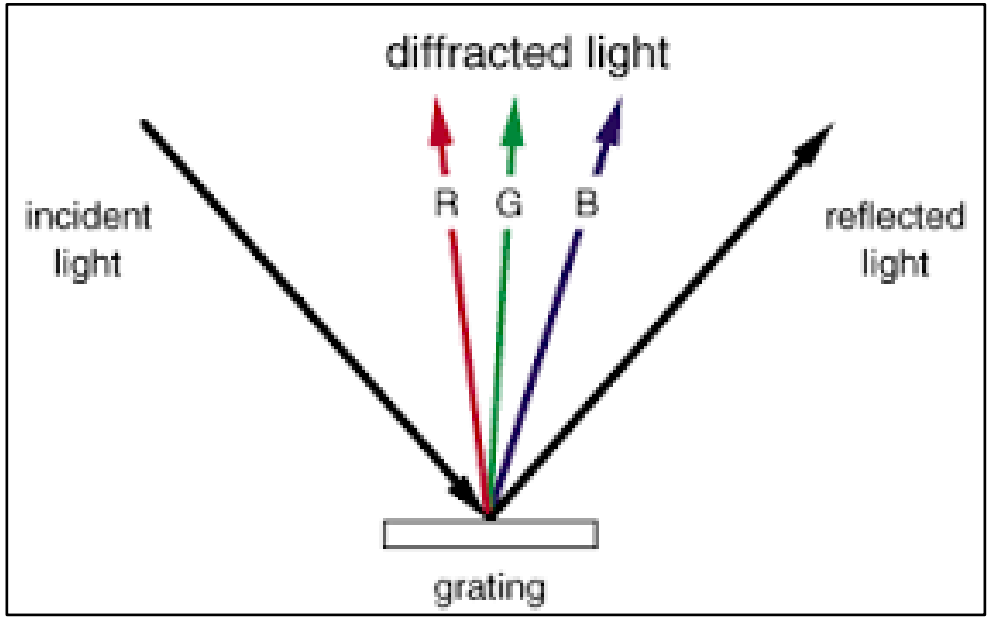
XRD provides information about phase identification, crystal size and crystal perfection, structural parameters (unit cell, edge, lengths) and degree of substitution of ions by other ions. The physical principles of characteristic X-ray generation are schematically illustrated in Figure



Schematic of X- ray Diffractometer

Diffraction is the slight bending of light as it passes around the edge of an object. The amount of bending depends on the relative size of the wavelength of light to the size of the opening. If the opening is much larger than the light's wavelength, the bending will be almost unnoticeable.

The change in direction of a wave, such as a light or sound wave, away from a boundary the wave encounters. **Reflected** waves remain in their original medium rather than entering the medium they encounter.



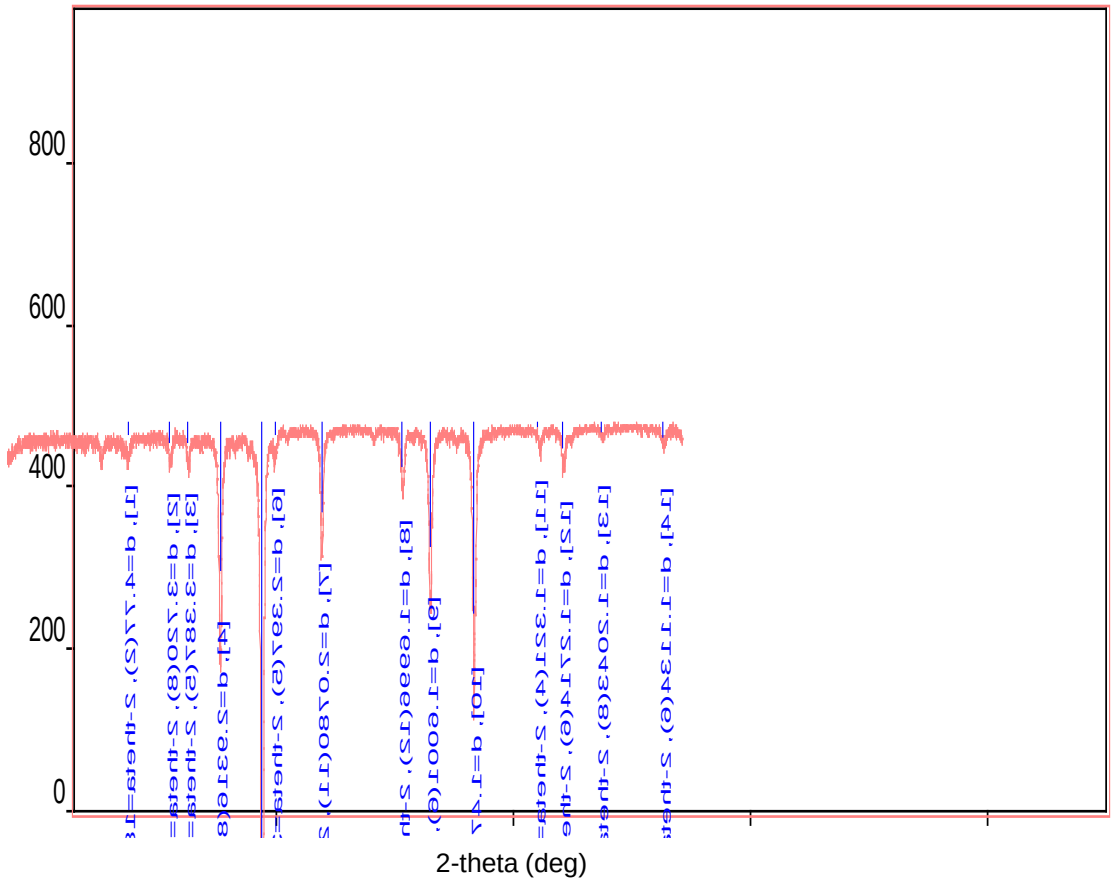
- The X-ray diffraction pattern of a powdered sample is a plot of the observed diffracted intensity of the X-rays against the Bragg angle, i. e. the angle at which the X-rays strike the crystal and for which the maximum interference is observed.

- An XRD pattern of a crystalline phase, therefore, consists of a number of reflections (peaks) of different intensities which provide a fingerprint of the atomic structure.

- From these patterns the mineral-specific distances between the atomic layers (d-values), can be calculated using the Bragg equation and from the set of d-values the mineral(s) can be identified.

- The X-ray pattern is preferably recorded stepwise using a Goniometer equipped with a proportional or scintillation counter and a registration unit. The Scherrer equation is employed to calculate the size of crystallites, in a diffraction pattern.

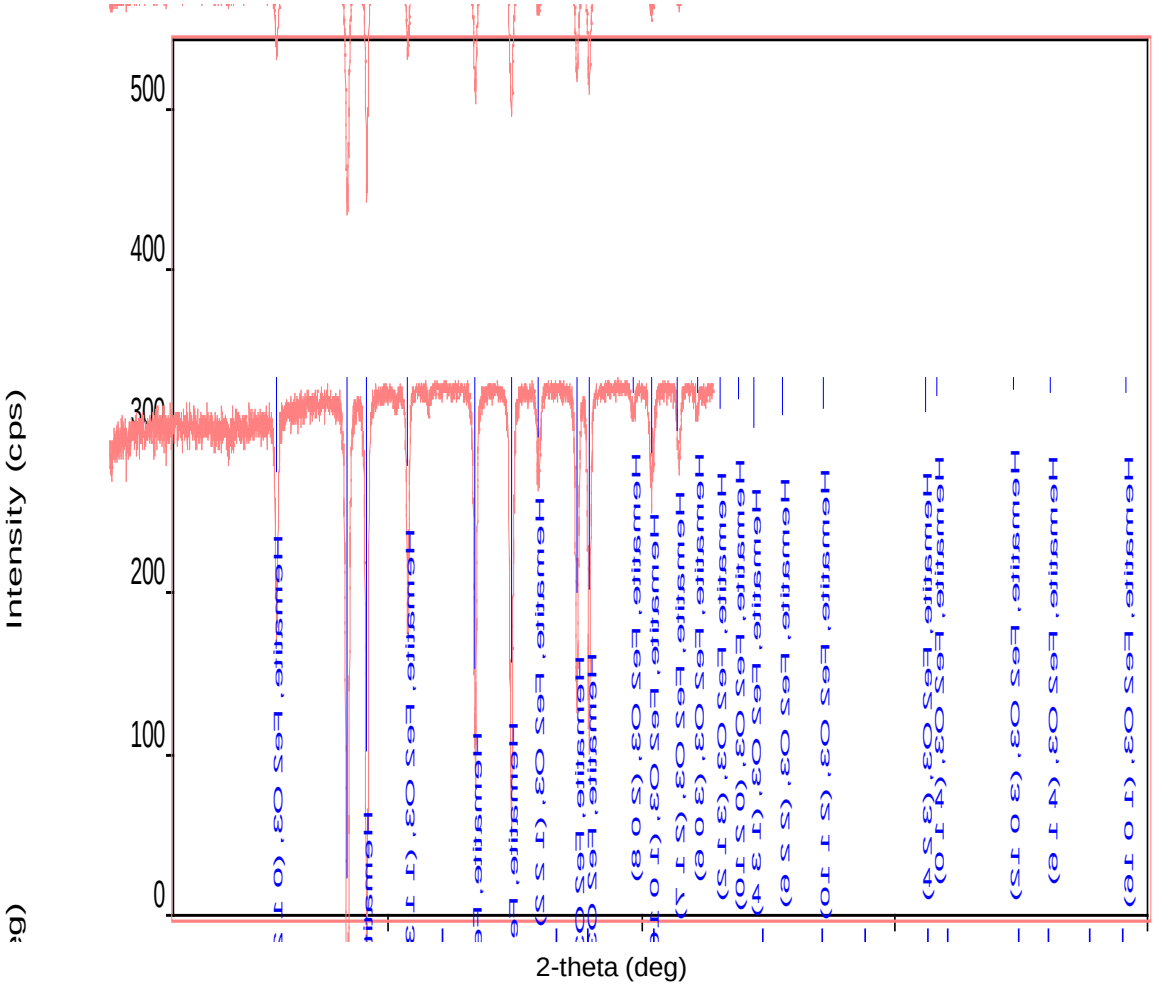
MAGHEMITE (γ-Fe_2O_3)



2-theta (deg)	d (ang.)	Height (cps)	Int. I(cps¥deg)	FWHM(deg)	Size	Phase name
18.58(10)	4.77(2)	13(4)	12.3(15)	0.70(13)	121(22)	Maghemite-C, syn, (1,1,1)
23.90(5)	3.720(8)	23(5)	10.9(10)	0.38(5)	221(26)	Maghemite-C, syn, (2,1,0)
26.29(4)	3.387(5)	22(5)	10.9(8)	0.46(3)	186(14)	Maghemite-C, syn, (2,1,1)
30.467(9)	2.9316(8)	180(13)	98.4(18)	0.393(12)	219(6)	Maghemite-C, syn, (2,2,0)
35.810(9)	2.5055(6)	540(23)	297(3)	0.387(8)	225(5)	Maghemite-C, syn, (3,1,1)
37.49(7)	2.397(5)	14(4)	6.4(12)	0.44(6)	200(28)	Maghemite-C, syn, (2,2,2)
43.52(2)	2.0780(11)	108(10)	57.5(11)	0.37(2)	239(13)	Maghemite-C, syn, (4,0,0)
53.90(4)	1.6996(12)	54(7)	28.7(11)	0.40(4)	233(22)	Maghemite-C, syn, (4,2,2)
57.55(2)	1.6001(6)	150(12)	82.0(16)	0.41(2)	231(13)	Maghemite-C, syn, (5,1,1)
63.154(19)	1.4710(4)	235(15)	131.0(18)	0.386(19)	252(12)	Maghemite-C, syn, (4,4,0)
71.3(2)	1.321(4)	4(2)	11.3(10)	2.0(4)	52(11)	Maghemite-C, syn, (5,3,2)
74.58(4)	1.2714(6)	31(6)	23.2(10)	0.58(4)	180(11)	Maghemite-C, syn, (5,3,3)
79.52(7)	1.2043(8)	11(3)	4.6(6)	0.34(7)	316(62)	Maghemite-C, syn, (4,4,4)
87.55(6)	1.1134(6)	15(4)	9.6(7)	0.55(7)	207(25)	Maghemite-C, syn, (6,4,2)

Phase name	Formula	Space group	ICDD
Maghemite-C, syn	Fe21.333 O32	212 : P4332 cubic	10830112 (ICDD)

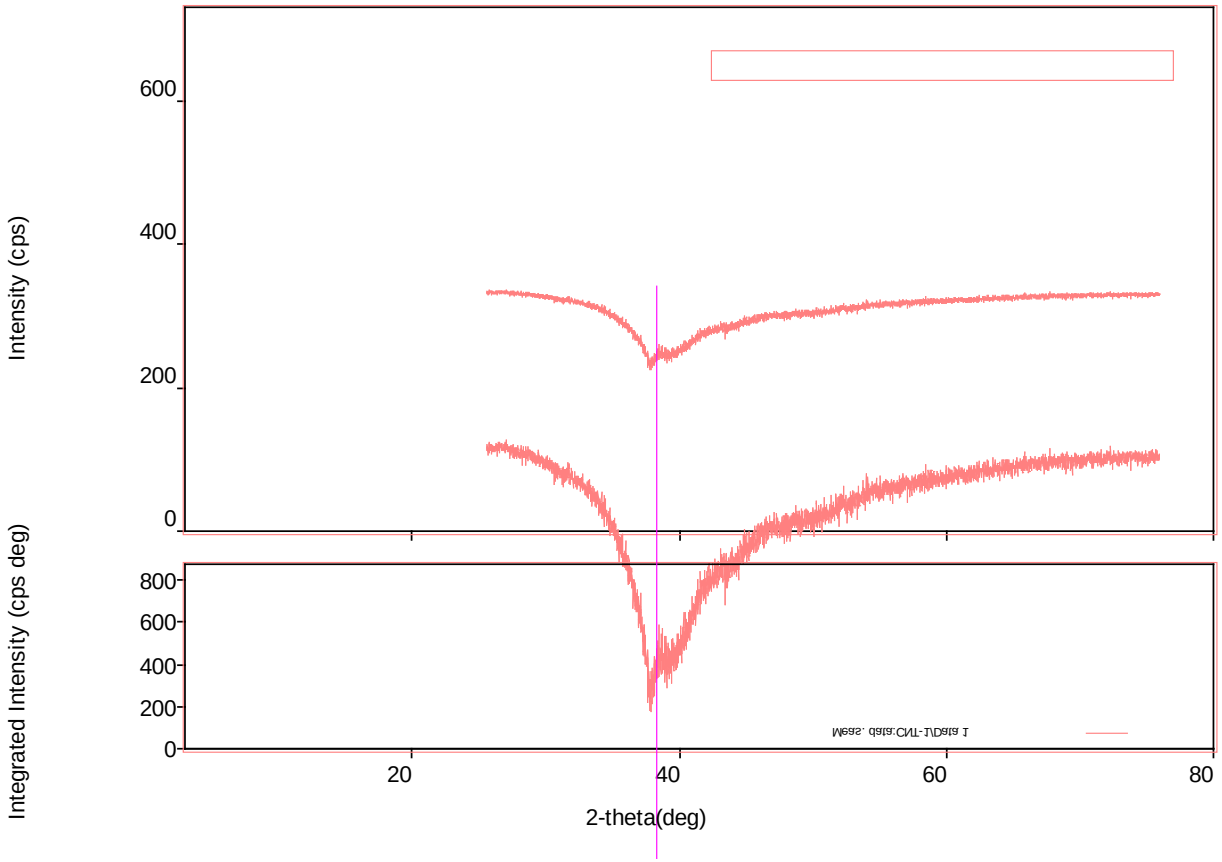
Hematite (α-Fe_2O_3)



2-theta (deg)	d (ang.)	Height (cps)	Int. I(cps¥deg)	FWHM(deg)	Size	Phase name
24.29(2)	3.661(3)	78(9)	37.3(12)	0.369(18)	230(11)	Hematite, (0,1,2)
33.319(9)	2.6869(7)	287(17)	149.9(18)	0.422(7)	205(3)	Hematite, (1,0,4)
35.782(9)	2.5074(6)	278(17)	108.5(15)	0.314(7)	277(6)	Hematite, (1,1,0)
41.012(19)	2.1989(10)	89(9)	39.2(9)	0.344(15)	258(12)	Hematite, (1,1,3)
49.67(2)	1.8341(7)	147(12)	70.8(14)	0.375(19)	244(12)	Hematite, (0,2,4)
54.231(15)	1.6900(4)	168(13)	87.2(14)	0.426(11)	219(6)	Hematite, (1,1,6)
57.81(4)	1.5936(10)	36(6)	19.5(9)	0.46(4)	207(19)	Hematite, (1,2,2)
62.60(2)	1.4826(4)	125(11)	60.0(13)	0.394(15)	246(9)	Hematite, (2,1,4)
64.191(17)	1.4497(3)	146(12)	61.8(11)	0.327(14)	299(13)	Hematite, (3,0,0)
69.75(8)	1.3471(13)	11(3)	6.1(5)	0.52(5)	194(20)	Hematite, (2,0,8)
72.04(3)	1.3099(5)	35(6)	22.8(9)	0.60(3)	170(7)	Hematite, (1,0,10)
75.62(4)	1.2564(6)	30(5)	14.7(7)	0.45(3)	234(15)	Hematite, (2,1,7)

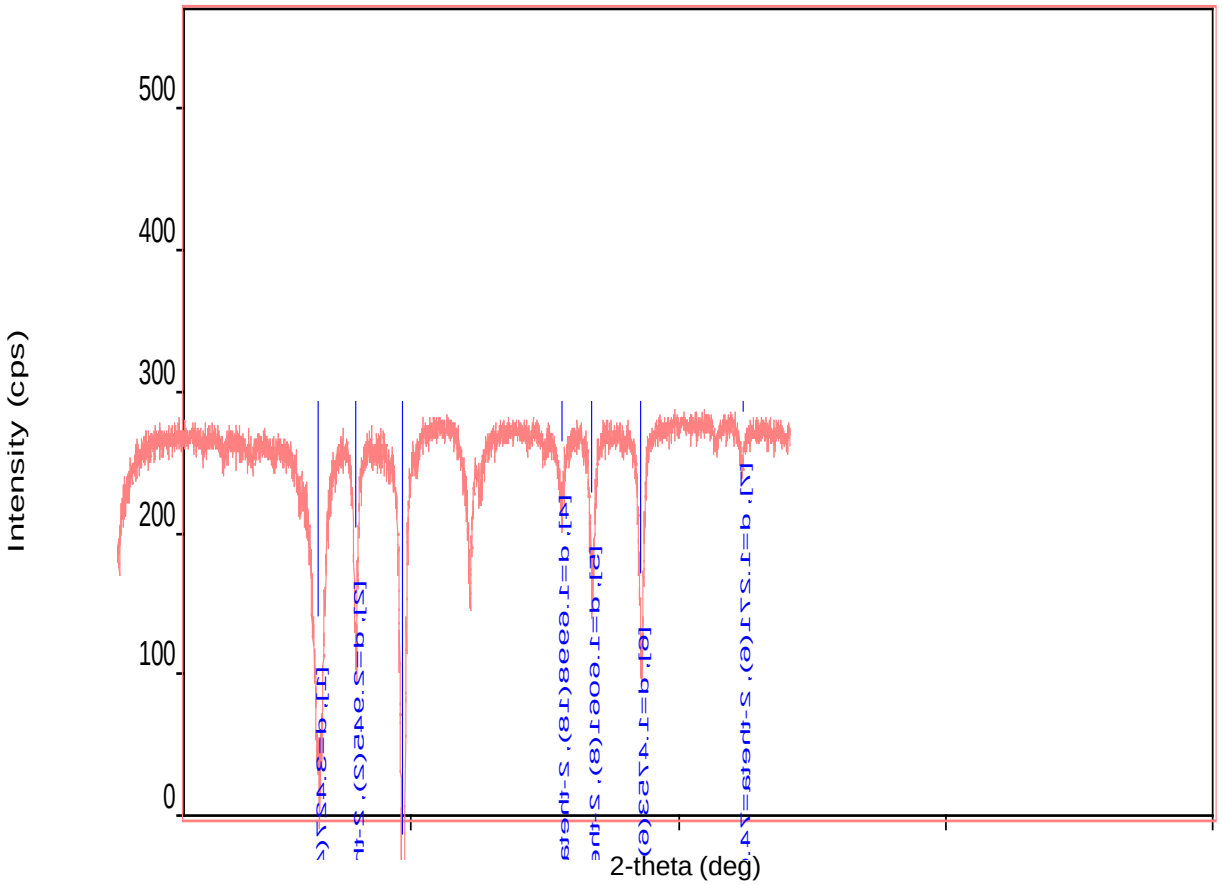
Phase name	Formula	Space group	ICDD
Hematite	Fe2 O3	167 : R-3c,hexagonal	11053 (ICDD)

Carbon Nanotubes (CNT)



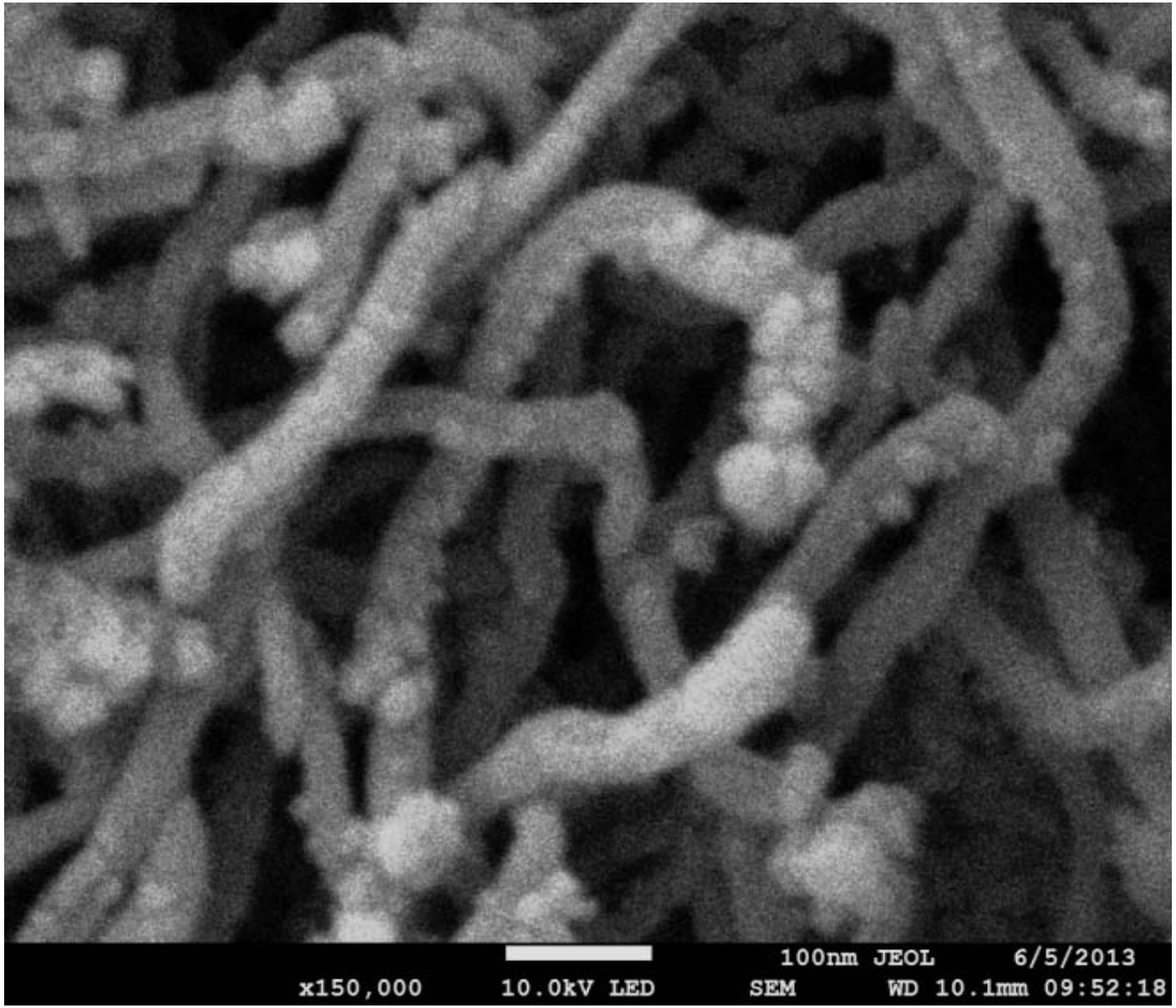
2-theta (deg)	d (ang.)	Height (cps)	Int. I(cps¥deg)	FWHM(deg)	Size	Phase name
26.47(6)	3.954(10)	232(15)	2795(15)	8.99(8)	9.41(8)	graphite (147 : P-3)

Maghemite decorated Carbon Nanotubes (CNT)



2-theta (deg)	d (ang.)	Height (cps)	Int. I(cps¥deg)	FWHM(deg)	Size	Phase name
25.98(3)	3.427(4)	149(12)	258(3)	1.33(3)	63.9(14)	Maghemite-C, syn, (2,1,1), Graphite, (0,0,2)
30.33(2)	2.945(2)	88(9)	55.9(16)	0.54(2)	158(6)	Maghemite-C, syn, (2,2,0)
35.662(16)	2.5156(11)	303(17)	246(3)	0.535(16)	163(5)	Maghemite-C, syn, (3,1,1)
53.89(6)	1.6998(18)	27(5)	28.6(16)	0.94(5)	99(5)	Maghemite-C, syn, (4,2,2), Graphite, (0,0,4)
57.32(3)	1.6061(8)	63(8)	44.4(18)	0.66(2)	144(5)	Maghemite-C, syn, (5,1,1)
62.95(3)	1.4753(6)	120(11)	97.6(16)	0.56(2)	175(8)	Maghemite-C, syn, (4,4,0)
74.6(4)	1.271(6)	5(2)	15(3)	2.3(4)	46(9)	Maghemite-C, syn, (5,4,0)

Phase name	Formula	Space group	ICDD
Maghemite-C, syn	Fe2 O3	213 : P4132	391346 (ICDD)
Graphite	C	147 : P-3	10640 (ICDD)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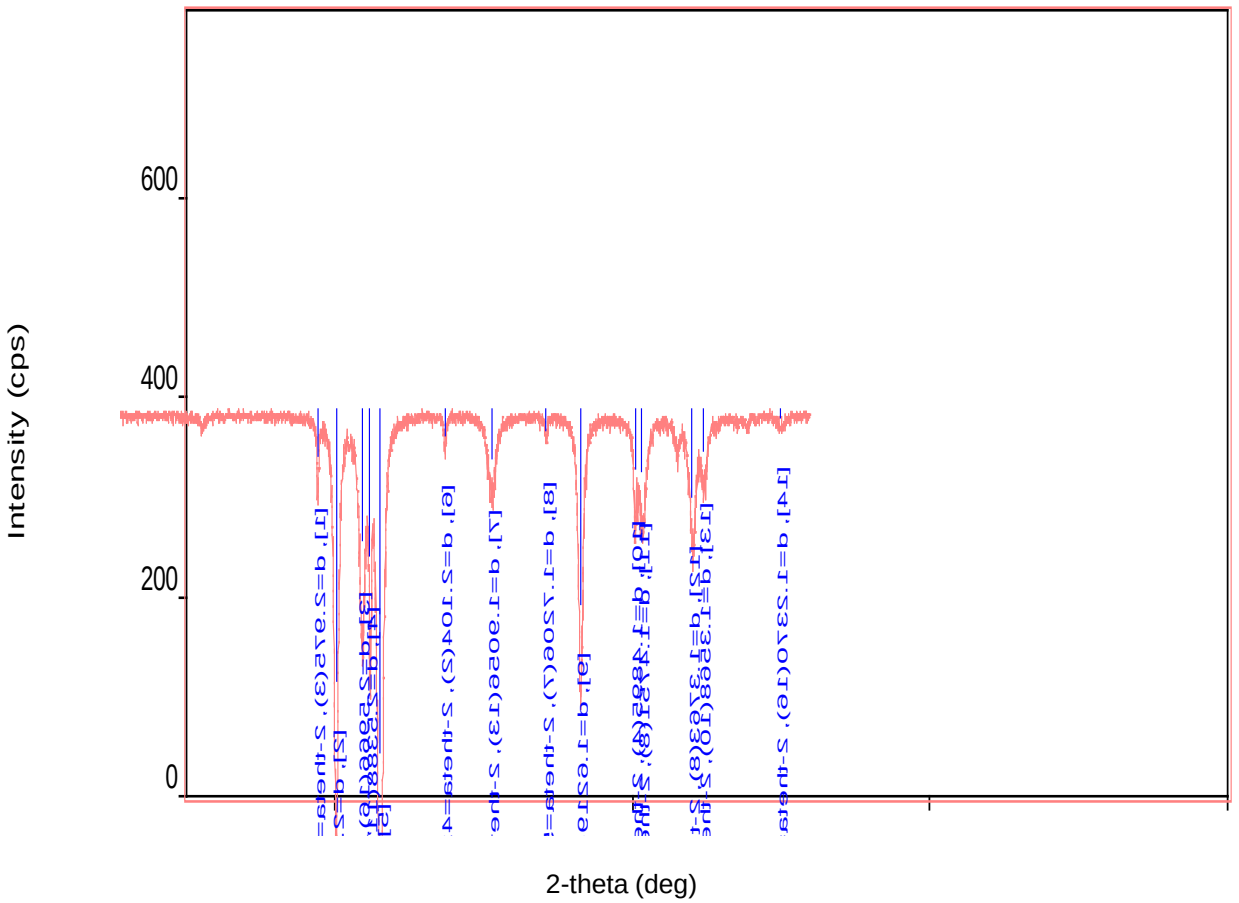
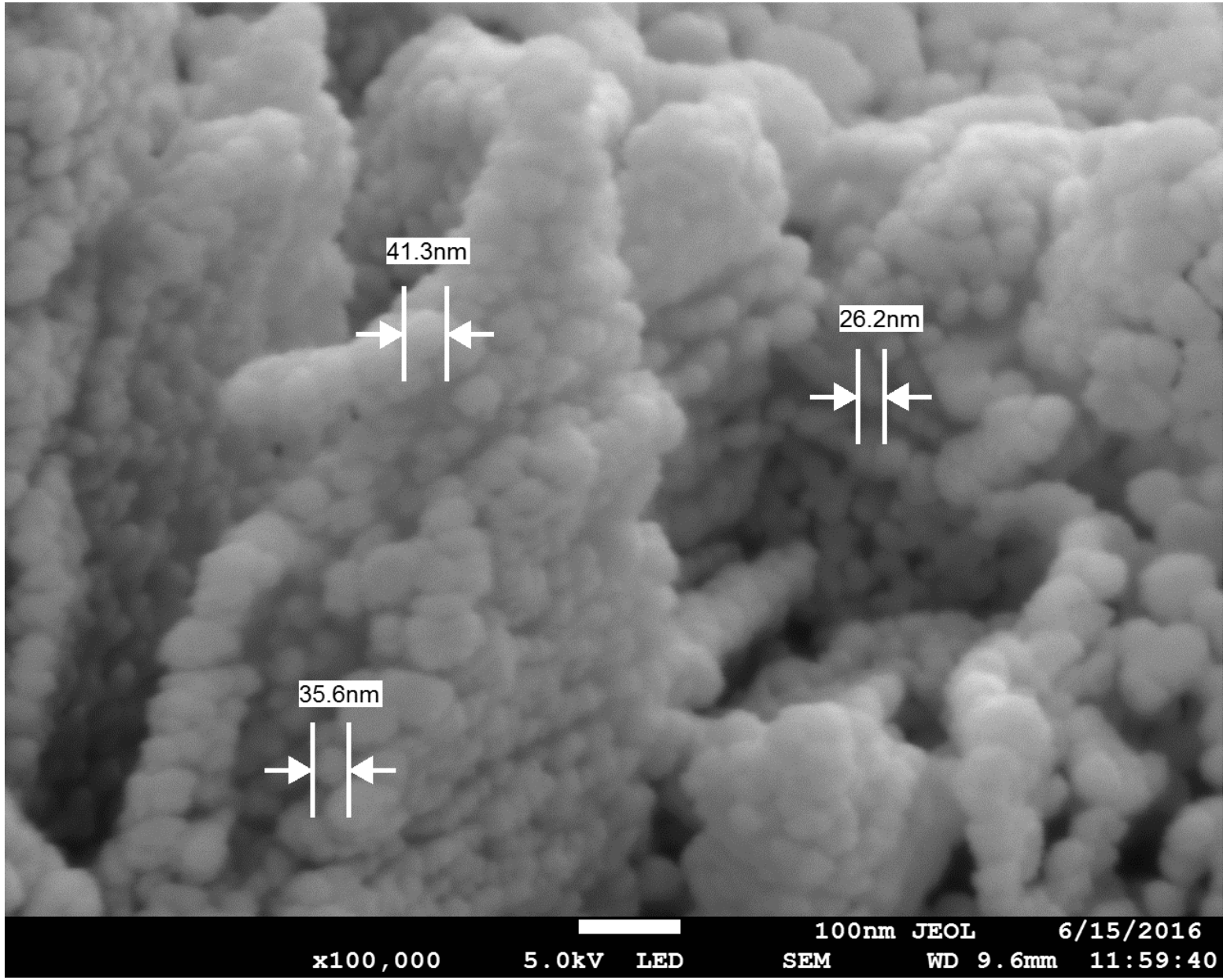
x150,000 10.0kV LED SEM WD 10.1mm 09:52:18

Hematite doped Zinc oxide

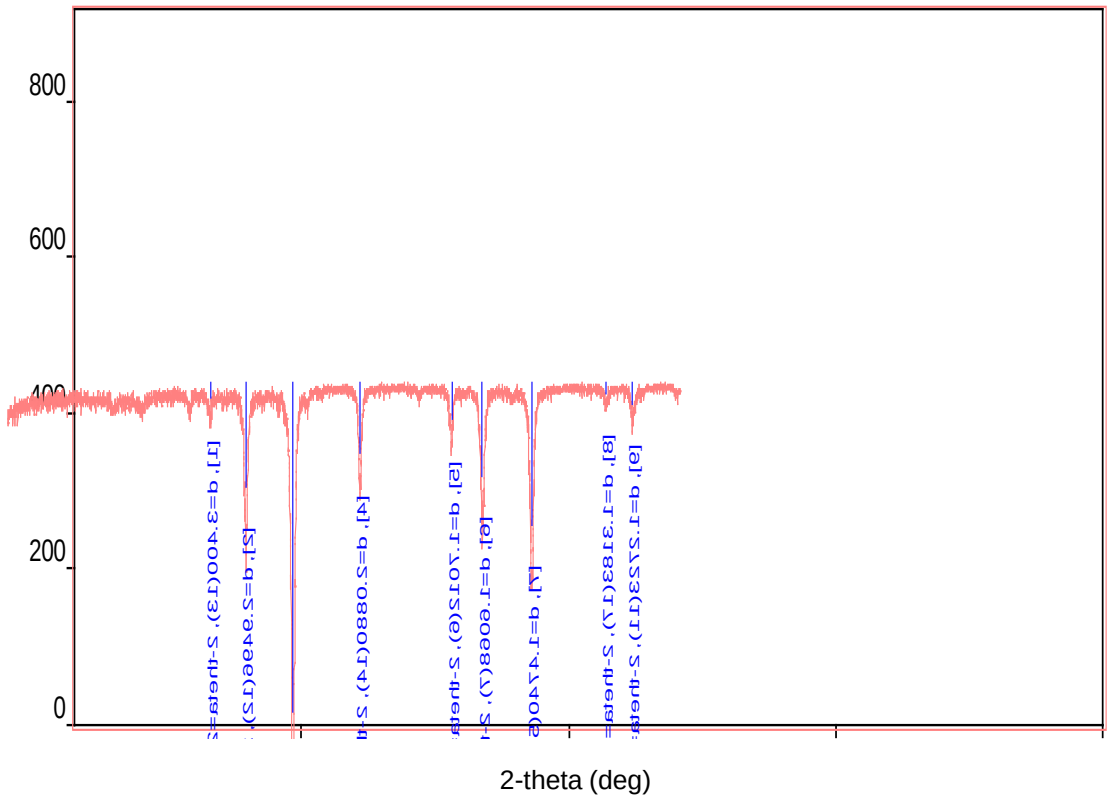


2-theta (deg)	d (ang.)	Height (cps)	Int. I(cps¥deg)	FWHM(deg)	Size	Phase name
30.01(3)	2.975(3)	48(7)	13.4(9)	0.23(3)	380(50)	zinc diiron(III) oxide, (2,2,0)
31.916(14)	2.8017(12)	277(17)	176(3)	0.499(12)	173(4)	Zinc Iron Oxide, (1,0,0)
34.51(2)	2.5966(16)	134(12)	115(3)	0.70(3)	125(5)	Zinc Iron Oxide, (0,0,2)
35.324(16)	2.5388(11)	150(12)	56(3)	0.304(17)	286(16)	zinc diiron(III) oxide, (3,1,1)
36.375(12)	2.4679(8)	352(19)	296(4)	0.687(10)	127.1(19)	Zinc Iron Oxide, (1,0,1), zinc diiron(III) oxide, (2,2,2)
42.96(5)	2.104(2)	26(5)	9.1(6)	0.25(5)	362(71)	zinc diiron(III) oxide, (4,0,0)
47.68(3)	1.9056(13)	51(7)	62.5(14)	0.99(3)	92(3)	Zinc Iron Oxide, (1,0,2), zinc diiron(III) oxide, (3,3,1)
53.19(2)	1.7206(7)	22(5)	5.6(5)	0.18(4)	516(112)	zinc diiron(III) oxide, (4,2,2)
56.71(2)	1.6219(5)	199(14)	145.8(18)	0.511(16)	184(6)	Zinc Iron Oxide, (1,1,0), zinc diiron(III) oxide, (5,1,1)
62.280(19)	1.4895(4)	61(8)	12.0(13)	0.18(2)	524(59)	zinc diiron(III) oxide, (4,4,0)
62.96(4)	1.4751(8)	63(8)	80(2)	1.20(4)	81(2)	Zinc Iron Oxide, (1,0,3)
68.07(4)	1.3763(8)	88(9)	72(5)	0.61(5)	164(12)	Zinc Iron Oxide, (1,1,2)
69.18(6)	1.3568(10)	42(6)	50(5)	0.88(11)	115(14)	Zinc Iron Oxide, (2,0,1)
77.03(12)	1.2370(16)	7(3)	6.4(7)	0.80(9)	133(16)	Zinc Iron Oxide, (2,0,2)

Phase name	Formula	Space group	ICDD
Zinc Iron Oxide	(Zn0.97 Fe0.03)0.989 O	186 : P63mc	10757918 (ICDD)
zinc diiron(III) oxide	Zn Fe2 O4	227 : Fd-3m,choice-2	10891009 (ICDD)

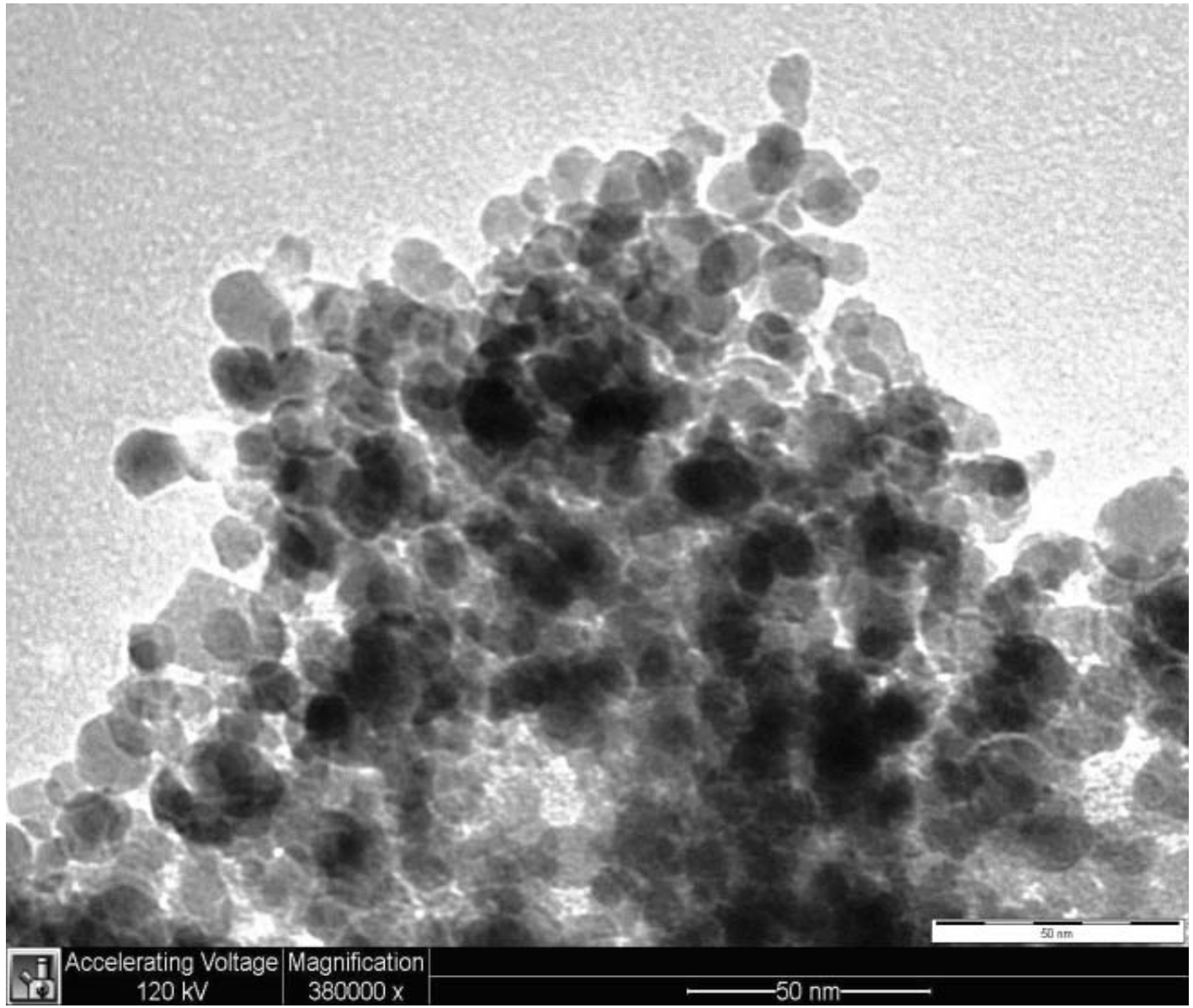

41.3nm
26.2nm
35.6nm
100nm JEOL 6/15/2016
x100,000 5.0kV LED SEM WD 9.6mm 11:59:40

Vanadium doped Maghemite



2-theta (deg)	d (ang.)	Height (cps)	Int. I(cps¥deg)	FWHM(deg)	Size	Phase name
26.19(10)	3.400(13)	22(6)	10.6(16)	0.37(9)	233(60)	Maghemite-C, syn, (2,1,1)
30.276(13)	2.9496(12)	148(16)	82(2)	0.395(17)	218(9)	Maghemite-C, syn, (2,2,0)
35.616(14)	2.5187(10)	474(28)	261(3)	0.368(14)	237(9)	Maghemite-C, syn, (3,1,1)
43.30(3)	2.0880(14)	101(13)	52.4(13)	0.36(3)	251(18)	Maghemite-C, syn, (4,0,0)
53.85(2)	1.7012(6)	52(9)	25.1(11)	0.41(2)	228(12)	Maghemite-C, syn, (4,2,2)
57.29(3)	1.6068(7)	134(15)	76.9(19)	0.45(2)	211(11)	Maghemite-C, syn, (5,1,1)
63.01(2)	1.4740(5)	203(18)	117(2)	0.42(3)	229(14)	Maghemite-C, syn, (4,4,0)
71.51(11)	1.3183(17)	14(5)	8.4(11)	0.52(7)	196(28)	Maghemite-C, syn, (6,2,0)
74.52(8)	1.2723(11)	30(7)	16.6(12)	0.46(6)	229(33)	Maghemite-C, syn, (5,3,3)

Phase name	Formula	Space group	ICDD
Maghemite-C, syn	Fe2 O3	213 : P4132	391346 (ICDD)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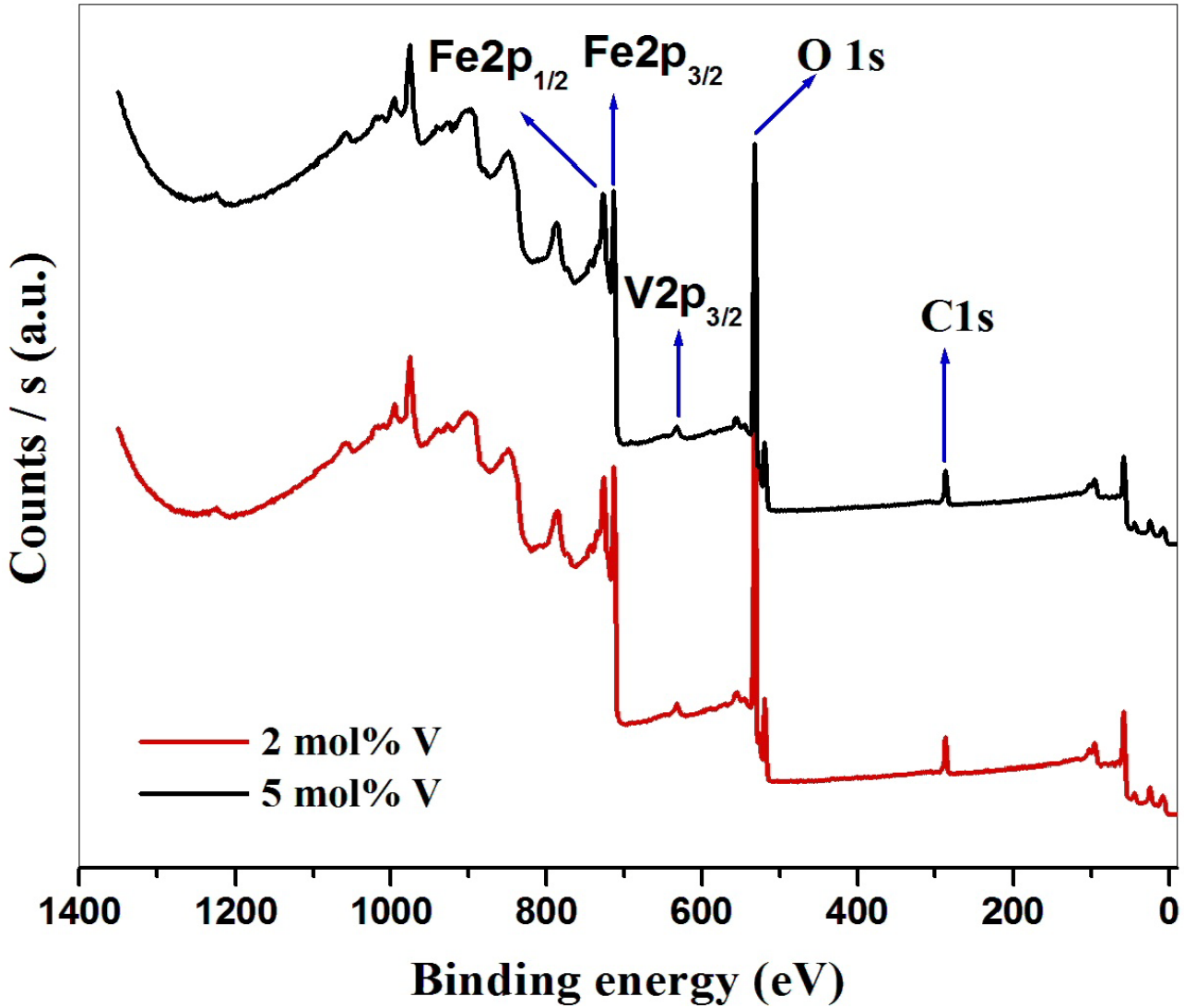
Accelerating Voltage
120 kV
Magnification
380000 x
50 nm



X-PS Spectroscopy Analysis of Vanadium doped Maghemite