Microstructural characterization, mechanical properties, electrical conductivity and corrosion studies of Fe-MWCNTs composite fabricated by conventional powder metallurgy

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Abstract

The present study reports the fabrication of MWCNTs reinforced (0.5, 1, 2, 4 vol. %) iron metal matrix composites by conventional powder metallurgy. The composites were fabricated by ultra-sonication for 3 hours followed by planetary milling of 20 min in a dual-drive planetary ball mill (DDPM) to obtain uniform dispersion of MWCNTs into iron matrix. The milled powders were then cold compacted and sintered at 1300 °C for 2 hours in Ar atmosphere. The milled composite powder and sintered composites were characterized by X-ray diffraction (XRD), Field emission scanning electron microscopy (FESEM), Transmission electron microscopy (TEM) and Raman spectroscopy. It has been observed that MWCNTs are stable, maintained their structure after milling and consolidation. The optimum density, hardness and compressive strength of 86 % relative density, 170 HV and 425 MPa respectively were found in Fe-4 vol. % MWCNTs composite. The non-lubricated sliding wear behaviour of the composites against a diamond ball was studied. The wear depth and possible wear mechanisms were investigated. The electrical conductivity of fabricated composites was evaluated and found that conductivity increases with increasing MWCNTs. The corrosion behaviour of the composites was studied in 3 wt. % NaCl solution. Fig. 1 (a) and (b) show the density and compressive stress-strain plots of Fe-MWCNTs composites.

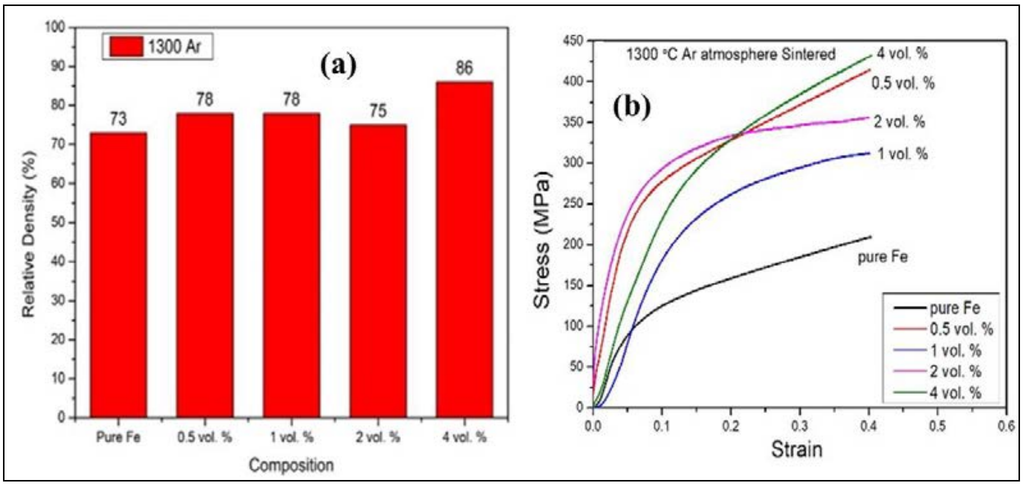

Fig. 1: (a) Relative density (b) Compressive stress-strain plot of Fe-MWCNTs composites

Fabrication of Fe-MWCNTs Composite by Conventional Powder Metallurgy

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Motivation

- Multiwall carbon nanotube is considered as a promising reinforcement due to its unique physical and mechanical properties.
- The combination of ultrasonic vibration followed by high energy planetary milling can provide uniform dispersions of MWCNTs in iron matrix.
- MWCNTs can be used as a good reinforcing agent for iron matrix due to its better load transfer at the interface.
- A good interface bonding may be obtained by vacant 3d orbital iron metal and p-orbital MWCNTs.
- Iron based composite reinforced with MWCNTs fabricated by powder metallurgy route can be projected for functional material.

Properties of Iron and MWCNTs

Matrix: Iron

- Ductile in nature, BCC structure
- Density - 7.2 g/cc
- Strength - 1.2 GPa
- Elastic modulus - 211 GPa
- Thermal Conductivity - 80.4 W/mK
- Conductivity $\sigma = 1.48 \times 10^6\, \Omega^{-1}\, m^{-1}$
- Melting point- 1539 ^oC

Reinforcement: MWCNTs

- Allotropy of Carbon, hexagonal structure
- Density- 2.1 g/cc
- Strength -100 GPa
- Elastic modulus - 1.28 TPa
- Thermal Conductivity - 6000 W/mK
- Electrical current density- 4×10^9 A/cm^2

Applications of MWCNT reinforced MMC:

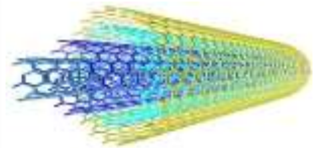
- ✓ Electronic packaging
- ✓ Sport industries
- ✓ Hydrogen storage
- ✓ Catalysis
- ✓ Aerospace
- ✓ Automobile
- ✓ Coating

Experimental Procedure

Milling Parameters:
Ball to powder wt. ratio- 10:1
Ball size-8, 10, 12 mm (stainless steel)
Vial volume- 1 L
Atmosphere- Argon
Milling time: 20 minutes

Ultrasonic vibration of MWCNTs for 3 hours

Planetary milling of 20 minutes



MWCNT

Electrolytic Fe Powder

Stainless steel jar

Dry milling

High Energy Dual-drive Planetary Ball Mill

Fe-0.5 vol.% MWCNTs

Fe-1 vol.% MWCNTs

Fe-2 vol.% MWCNTs

Fe-4 vol.% MWCNTs

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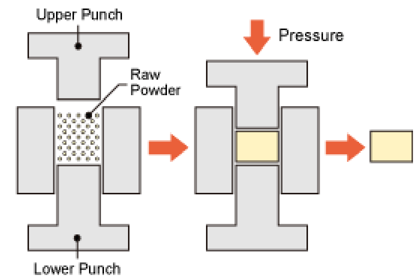
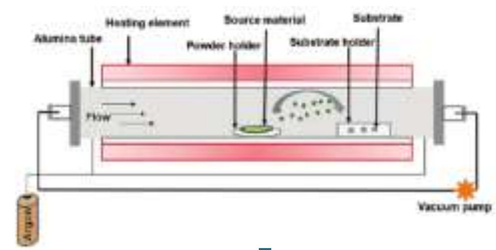
Fe- MWCNTs Composite Powder

After milling

Milled Powder Characterization:
- XRD Analysis
- FESEM with EDS analysis
- TEM



Cold Compaction, 500 MPa pressure

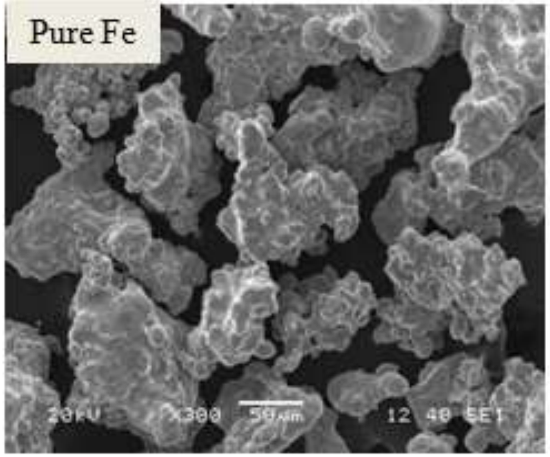


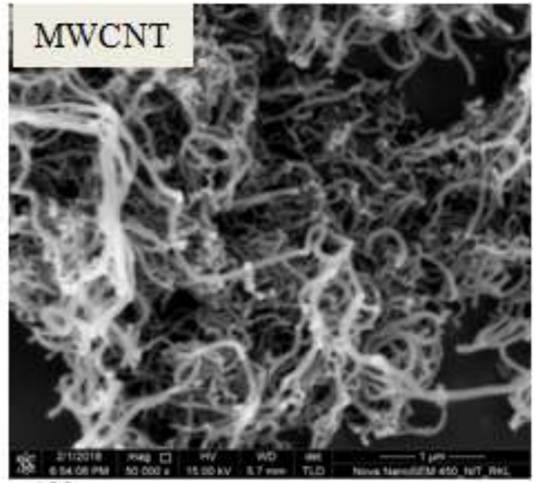
Conventional Sintering, 1300 °C, 2 h, Ar gas

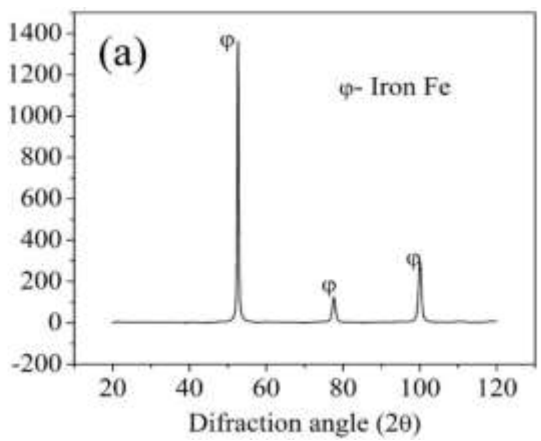
Sintered Composite

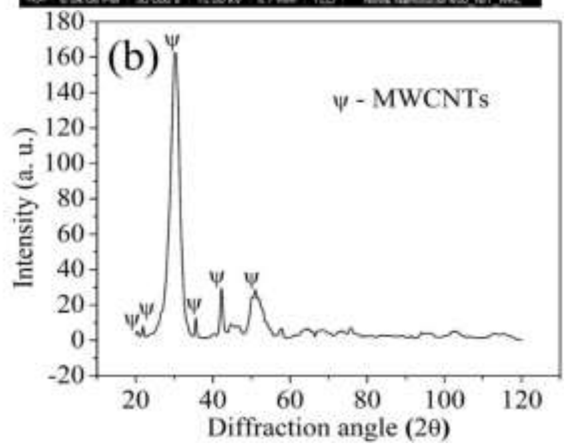
- XRD Analysis
- Microstructure (SEM)
- Sintered Density
- Vickers Hardness
- Compression Test

Initial Powder Characterization



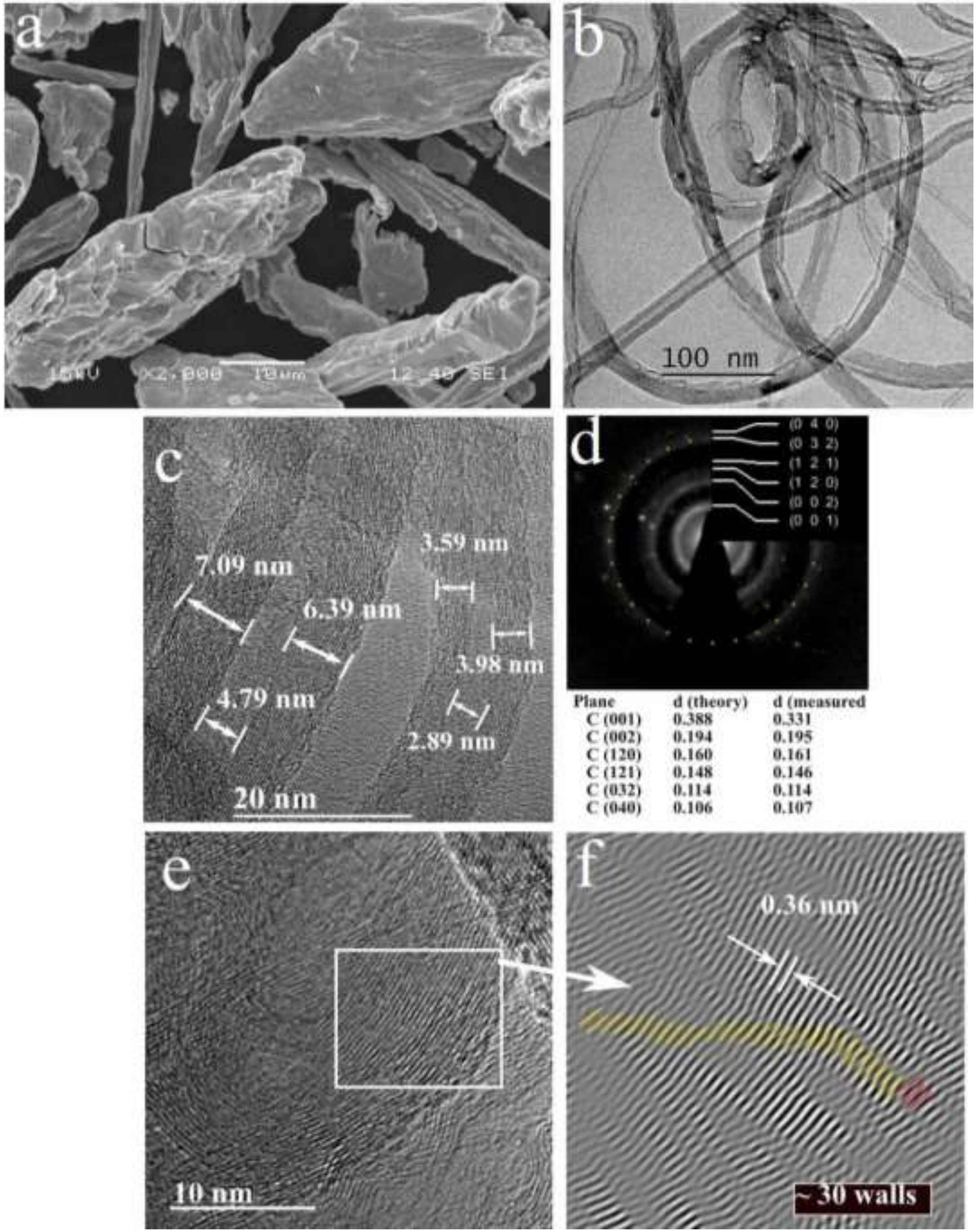




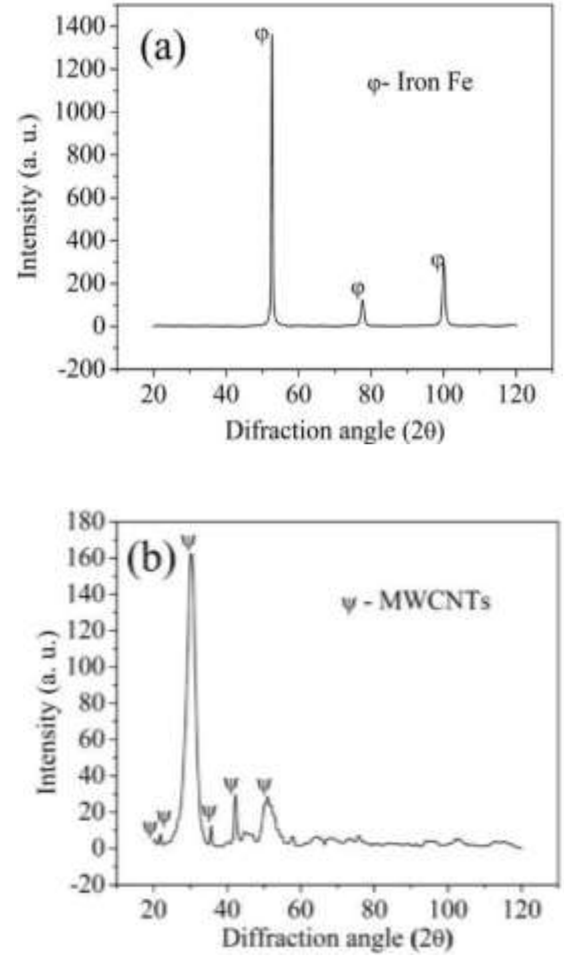


Sample name	Bulk density (g/cc)	Tap density (g/cc) after 2000 taps	Flowability/ angle of repose
Pure Fe	3.27	3.99	6°
Pure MWCNT	0.037	0.063	31°

- Electrolytic iron powder shows sponge shape
- MWCNTs are highly agglomerated and tube diameter <100 nm
- Flowability of iron powder is good, whereas flowability of MWCNTs is poor
- Tap density of Fe and MWCNTs are higher than bulk density

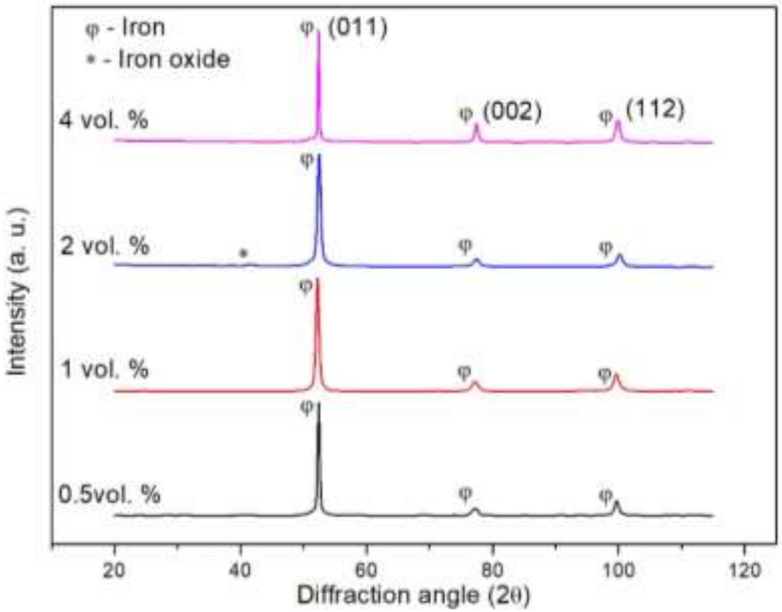


SEM micrograph of (a) iron powder and (c-f) HRTEM micrographs of MWCNTs

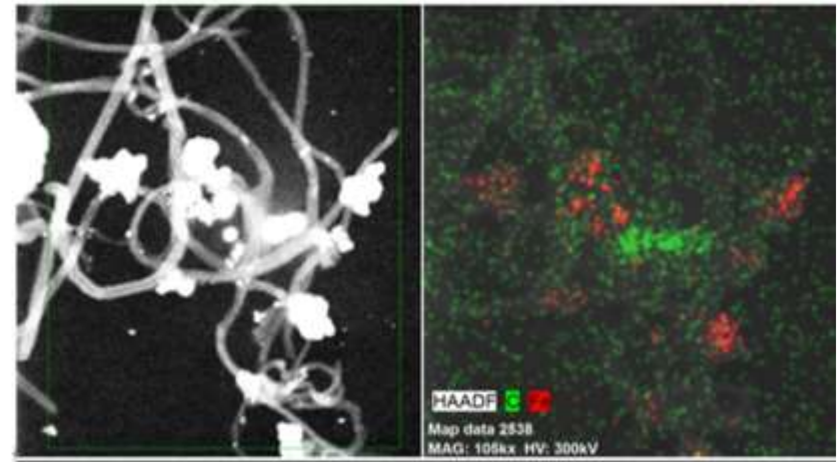


XRD spectra of (a) iron powder and (b) MWCNTs

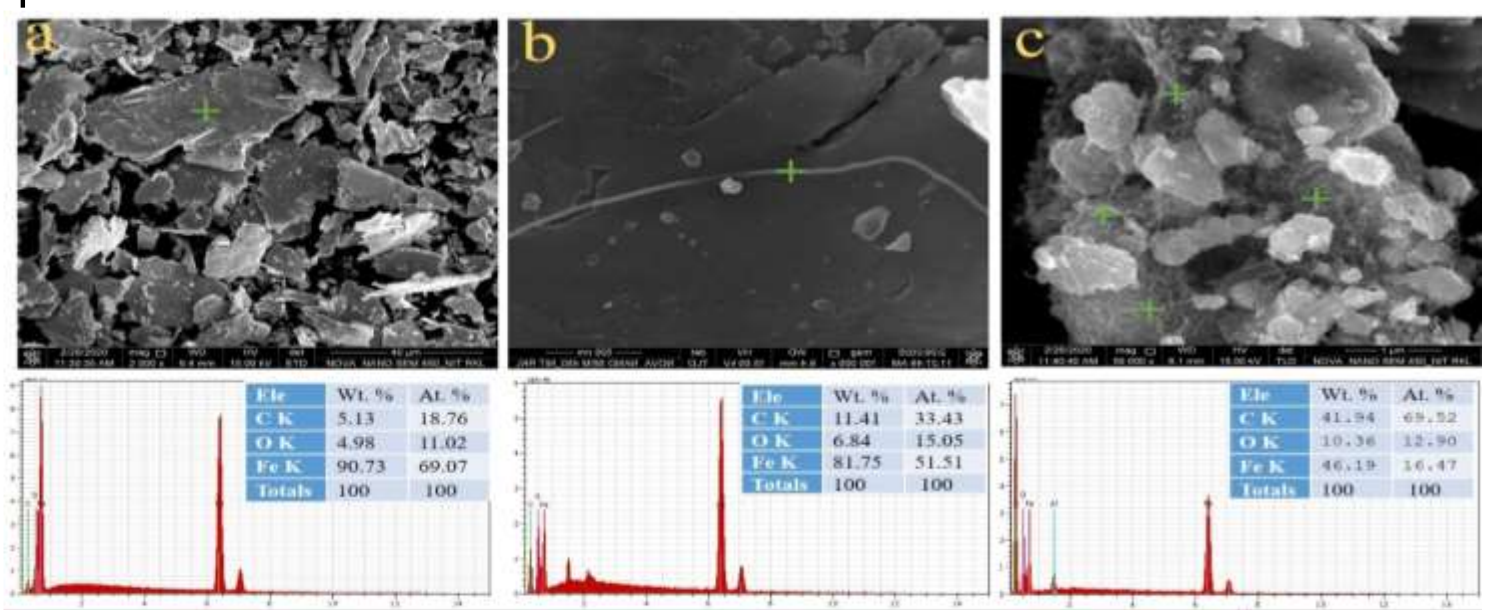
Characterization of Powder



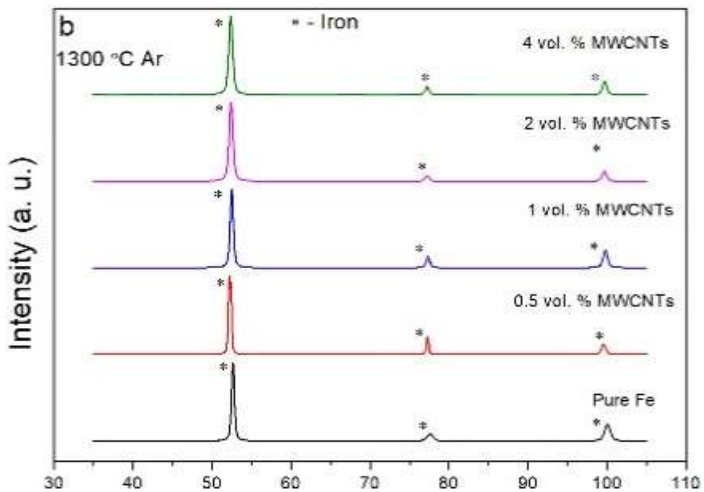
XRD spectra of Fe-MWCNTs composites powder



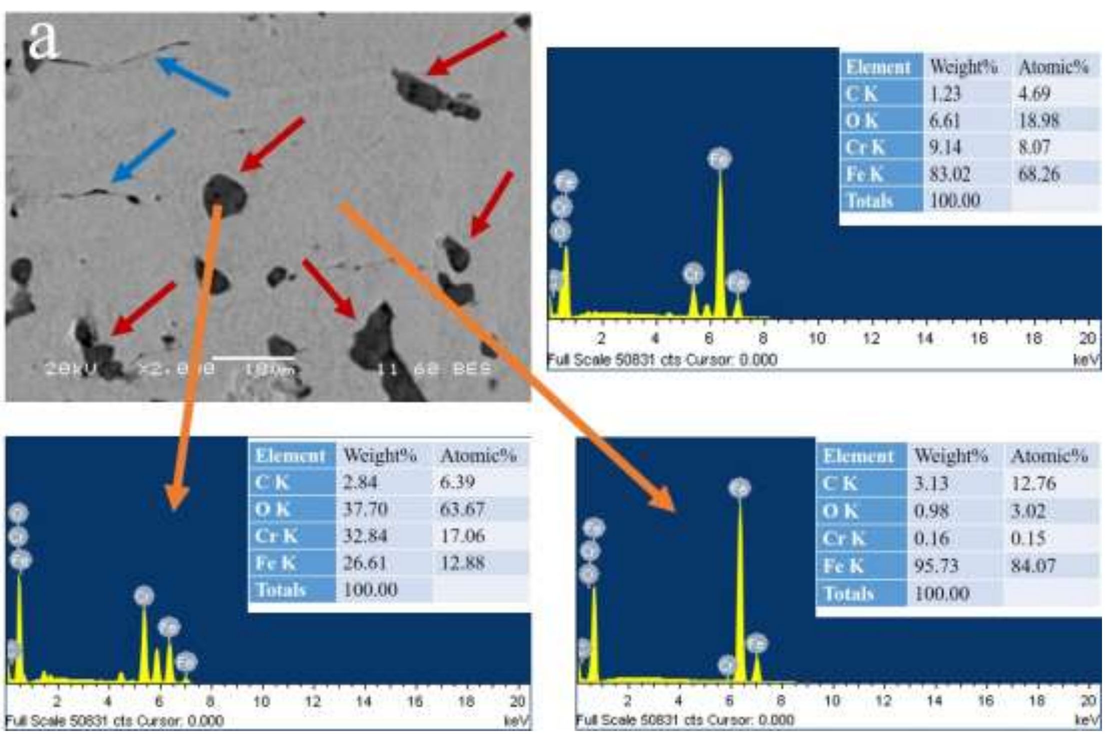
TEM micrograph, elemental mapping and elemental analysis of Fe-2 vol. % MWCNTs composite powder



Characterization of Sintered Composites



- Peaks of only iron are visible after consolidation at 1300 C for 2 h in Ar atmosphere.
- No other phases are formed during consolidation.
- Peaks of MWCNTs are not visible due to low crystallinity as compared to iron.

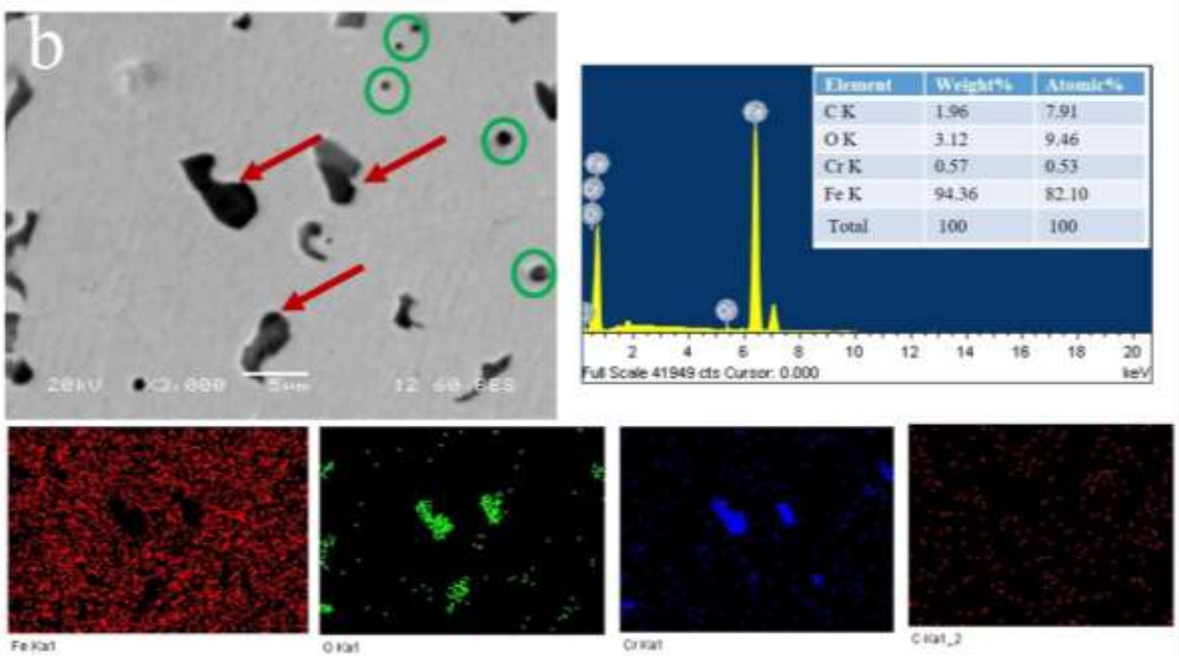


Element	Weight%	Atomic%
C K	1.23	4.69
O K	6.61	18.98
Cr K	9.14	8.07
Fe K	83.02	68.26
Totals	100.00	

Element	Weight%	Atomic%
C K	2.84	6.39
O K	37.70	63.67
Cr K	32.84	17.06
Fe K	26.61	12.88
Totals	100.00	

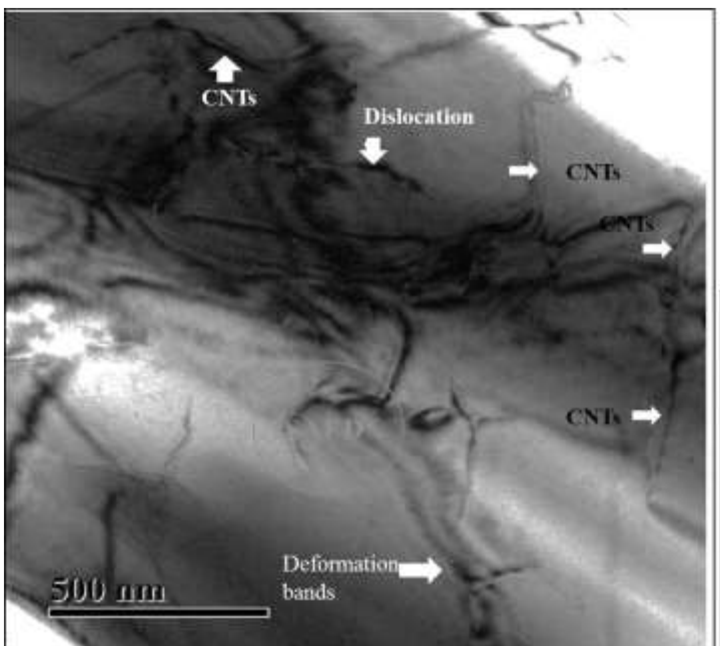
Element	Weight%	Atomic%
C K	3.13	12.76
O K	0.98	3.02
Cr K	0.16	0.15
Fe K	95.73	84.07
Totals	100.00	

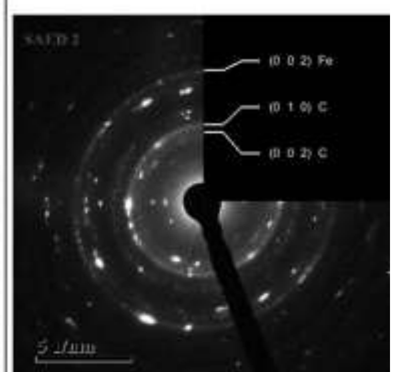
- There is precipitation (black) of iron oxide and Cr into iron oxide.



Element	Weight%	Atomic%
C K	1.96	7.91
O K	3.12	9.46
Cr K	0.57	0.53
Fe K	94.36	82.10
Total	100	100

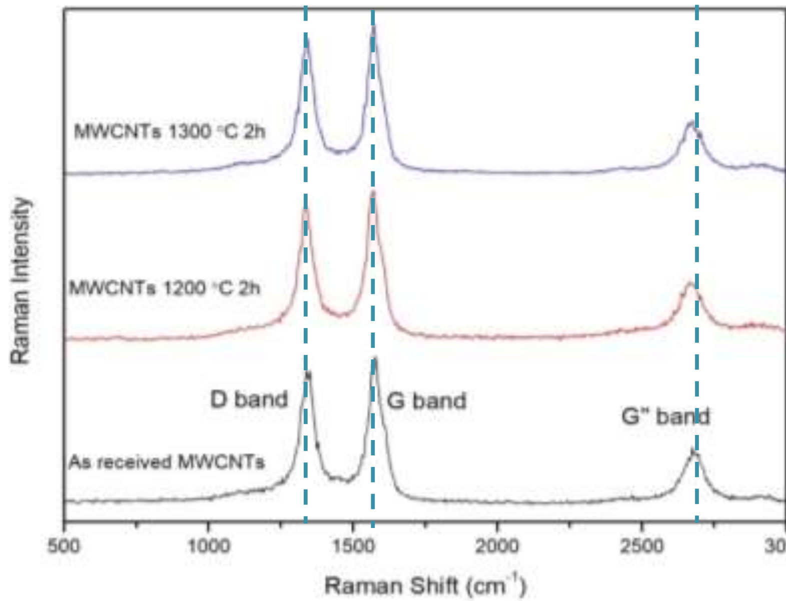
- Isolated spherical pores represent completion of sintering.
- MWCNTs are uniformly distributed throughout the matrix and also into second phase precipitates.
- Black colour precipitates mainly contain iron oxide and Cr.



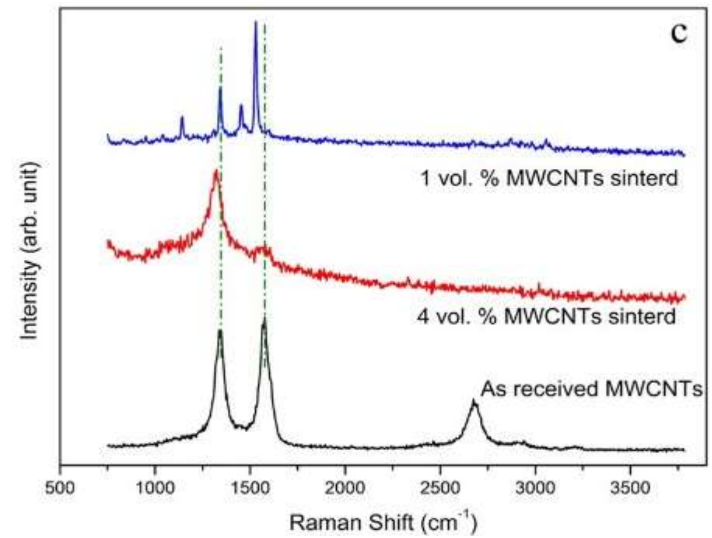


- BF TEM micrograph shows the presence of MWCNTs and dislocations.
- SAD pattern shows rings of iron and carbon.

Stability of MWCNTs



Raman spectra of as received MWCNTs, MWCNTs heated at 1200 and 1300 °C under Ar gas at 1300 °C for 2 hours

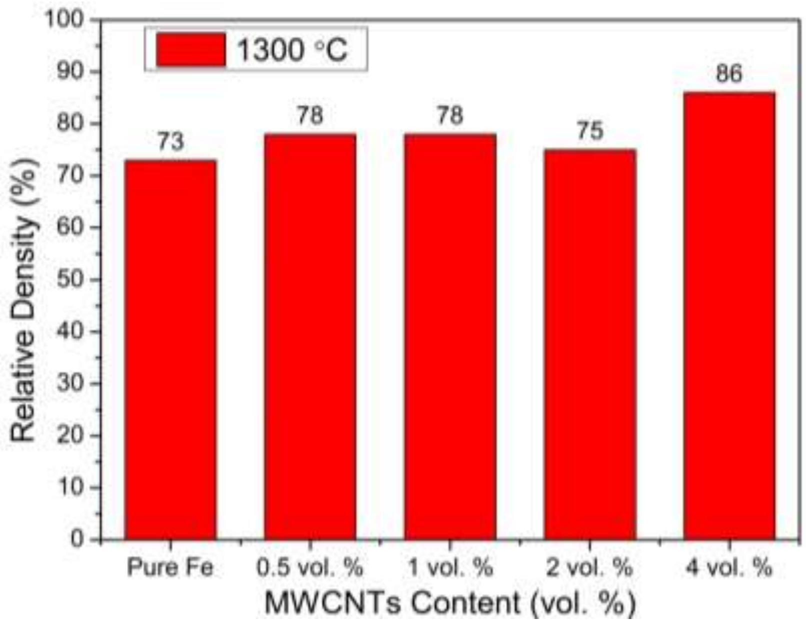


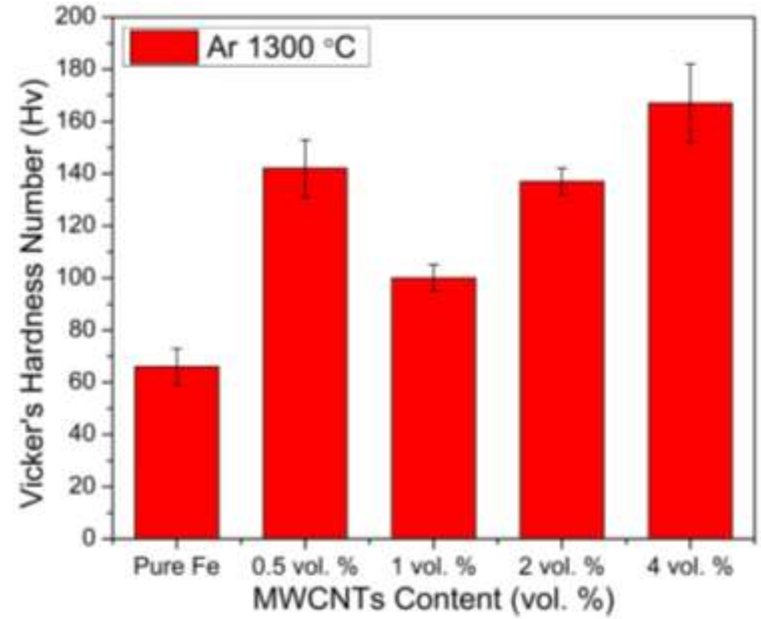
Raman spectra of pure as received MWCNTs, Fe-1 vol. % MWCNTs and Fe-4 vol. % MWCNTs composites sintered at 1300 °C in argon gas.

- MWCNTs are stable at 1300 °C.
- D, G and G" bands are clearly visible .
- D, G and G" are positioned at 1341, 1573 and 2700 cm^{-1}

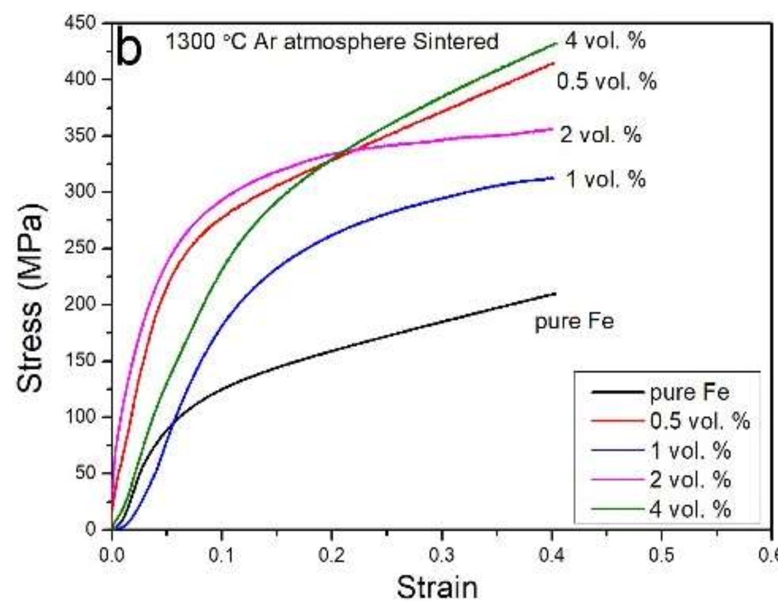
Sample	Raman Peak Shift (cm^{-1})		I_D/I_G
	D band	G band	
Pure MWCNTs	1344	1578	0.96
Fe-4 vol. % MWCNTs 20 min milling	1341	1577	1.04
Fe-4 vol. % MWCNTs 1300 °C Ar Sintering	1330	1560	1.07
Fe-1 vol. % MWCNTs 1300 °C Ar Sintering	1310	1560	1.07

Properties Evaluation





- Maximum density of 86 % (relative density) and Vickers hardness of 170 VHN are obtained for the composite consolidated at 1300 °C under Ar for 2 hours.
- The maximum compressive strength of 430 MPa is obtained for Fe-4 vol. % MWCNTs MMC sintered in Ar at 1300 °C for 2 hours.



Strengthening mechanisms

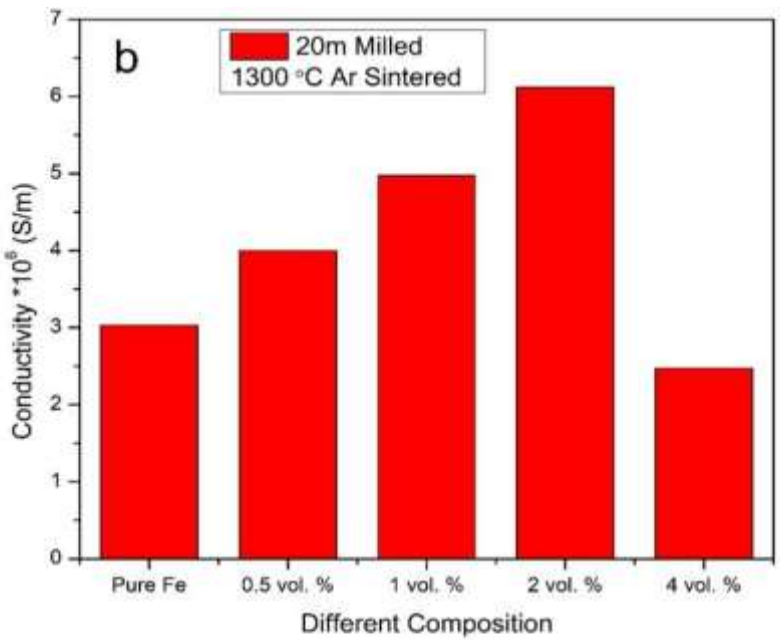
- **Dislocation strengthening:** large difference in elastic modulus (EM) and coefficient of thermal expansion between matrix (Fe) and reinforcement (MWCNTs) CTE

$$\Delta\sigma_{EM} = \sqrt{3}\alpha G b \sqrt{\frac{6 v_p \varepsilon}{b d_p}}$$

$$\Delta\sigma_{CTE} = \sqrt{\beta} G b \sqrt{\frac{12 v_p}{b d_p (1-v_p)} \Delta C \Delta T}$$

Where α, β are constants, G-shear modulus of pure iron, b-Burgers Vector of pure iron, v_p-particle volume fraction, $d_p - mean\ particle\ dia.$, ε-uniform deformation, ΔT-temperature difference between consolidation and room temperature, ΔC-difference in coefficient of thermal expansion

- **Precipitation strengthening:** Cr, iron oxides and MWCNTs precipitates as second phases at the particle boundaries and inside the matrix during consolidation (black coloured)
- **Load transfer:** Transfer of load from matrix to reinforcements (MWCNTs) due to high aspect ratio of MWCNTs



- Electrical conductivity of Fe-MWCNTs increases up to addition of 2 vol. % MWCNTs, after that it decreases. The maximum electrical conductivity of 6.5×10^6 S/m has been achieved in 2 vol. % MWCNTs reinforced composite.

Electrical conductivity of Fe-MWCNTs composites

Conclusions

- Fe-MWCNTs metal matrix composites were successfully fabricated by powder metallurgy route.
- MWCNTs are stable both after milling and sintering in Ar atmosphere at 1300 °C.
- Ultra sonication followed by high energy planetary for 20 minutes can be used for homogeneous mixing of Fe and MWCNTs.
- Fe-4 vol. % MWCNTs MMC consolidated at 1300 °C in Ar exhibits optimum 86 % relative density, hardness of 170 VHN and compressive strength of 430 MPa.
- Electrical conductivity of Fe-MWCNTs increases up to 2 vol. % of MWCNTs, after that it decreases. The maximum electrical conductivity of 6.5×10^6 S/m has been achieved in 2 vol. % MWCNTs reinforced composite.

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