Si_3N_4 Ceramic Composite with the addition of MWCNTs

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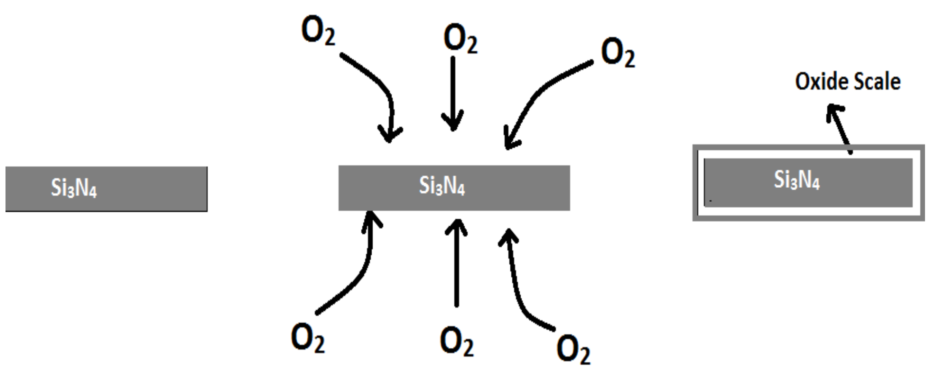
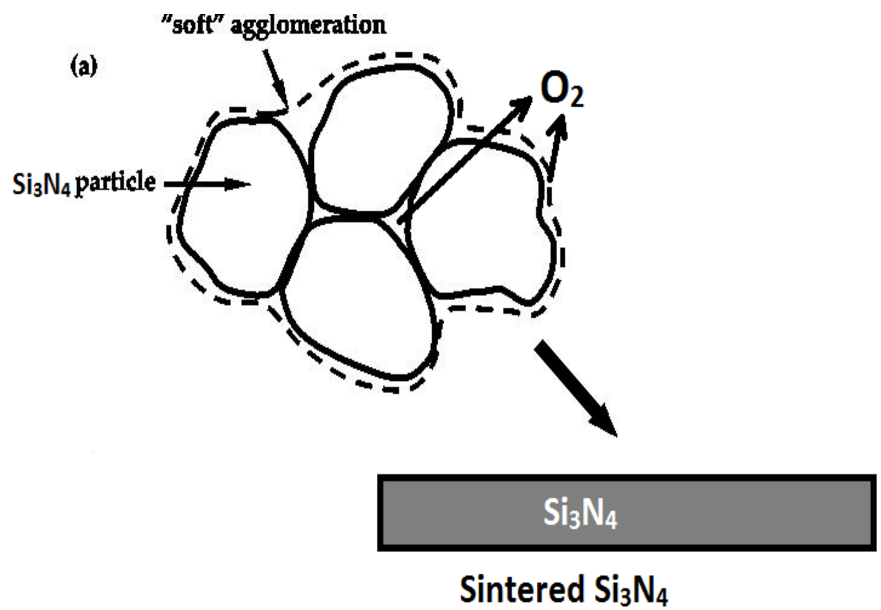
Aim of work

- To study the effect of oxidized Si_3N_4 powder particles on the mechanical properties of hot isostatic pressed Si_3N_4–CNTs/Grpahene composite material.





Plans Accomplished

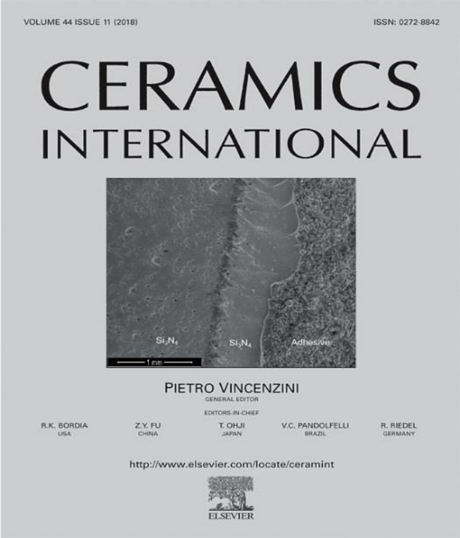
1st Semester

Si_3N_4		
1434	Si_3N_4 (Ref.)	
1435	Si_3N_4 (10 hrs Oxidized)	
1436	Si_3N_4 (20 hrs Oxidized)	

2nd Semester

Addition of 1% CNTs and 2% Graphene		
1459	Si_3N_4 - 1% CNTs – 2% Graphene (Ref.)	
1460	Si_3N_4 (10 hrs oxidized) – 1% CNTs – 2% Graphene	
1461	Si_3N_4 (20 hrs oxidized) – 1% CNTs – 2% Graphene	
Addition of 3% CNTs		
1462	Si_3N_4 – 3% CNTs (Ref.)	
1463	Si_3N_4 (10 hrs oxidized) – 3% CNTs	
1464	Si_3N_4 (20 hrs Oxidized) – 3% CNTs	
Addition of 3 wt.% Graphene		
1468	Si_3N_4 – 3% Graphene (Ref.)	
1469	Si_3N_4 (10 hrs Oxidized) – 3% Graphene	
1470	Si_3N_4 (20 hrs oxidized) – 3% Graphene	

Progress Report 2017-2018



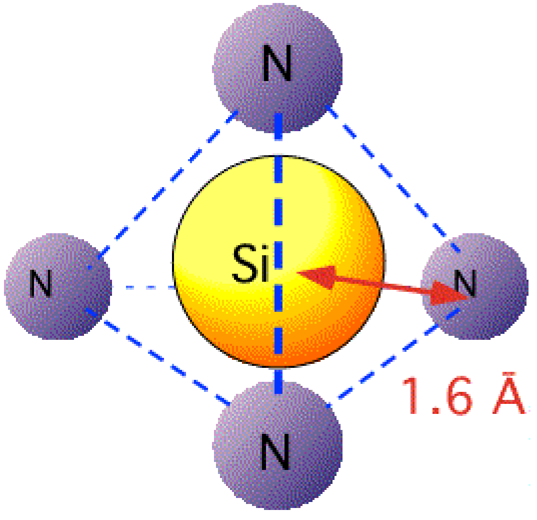
- **A. Qadir,** Z. Fogarassy, Z. E. Horváth, K. Balazsi, and C. Balazsi, "Effect of the oxidization of Si3N4 powder on the microstructural and mechanical properties of hot isostatic pressed silicon nitride," *Ceramics International*, vol. 44, no. 12, pp. 14601–14609, Aug. 2018. (Impact Factor 2.986). https://doi.org/10.1016/j.ceramint.2018.05.081
- Awais Qadir; Katalin Balazsi; Csaba Balazsi, **Fabrication of silicon nitride/MWCNTs composite from oxidized α-Si_3N_4 starting powder by hot isostatic pressing technique, Materials Technology, June 2018 (to be submitted)** (Impact Factor 1.23).

Participation in Conferences 2017-2018

- Junior EuroMat Conference 2018, Budapest
- Fine Ceramics Day 2018, Budapest
- Attended Hungarian Microscopic Conference 2017 in Siofok, Hungary
- Poster Presentation in ECerS 2017, 15th Conference & Exhibition of the European Ceramic Society, 2017
- Poster Presentation International Conference Deformation and Fracture in PM Materials, High Tatras, 2017. Oct.22-25.
- Poster Presentation in Joint ICTP-IAEA Workshop on Fundamentals of Vitrification and Vitreous Materials for Nuclear Waste Immobilization, The Abdus Salam Centre for Theoritical Physics (ICTP), Trieste Italy. Nov. 06 -10, 2017.
- Oral Presentation "17th PhD Students Materials Science Day", University of Pannon, Veszprem, Hungary, Dec. 4. 2017
- Doctoral Summer School at Károly Róbert University, August 2017

Silicon nitride

- Silicon nitride (Si_3N_4) based ceramics are gaining more attention due to their promising high-temperature thermal and mechanical properties.
- Three crystallographic structures of silicon nitride (Si_3N_4), α, β and γ phases.



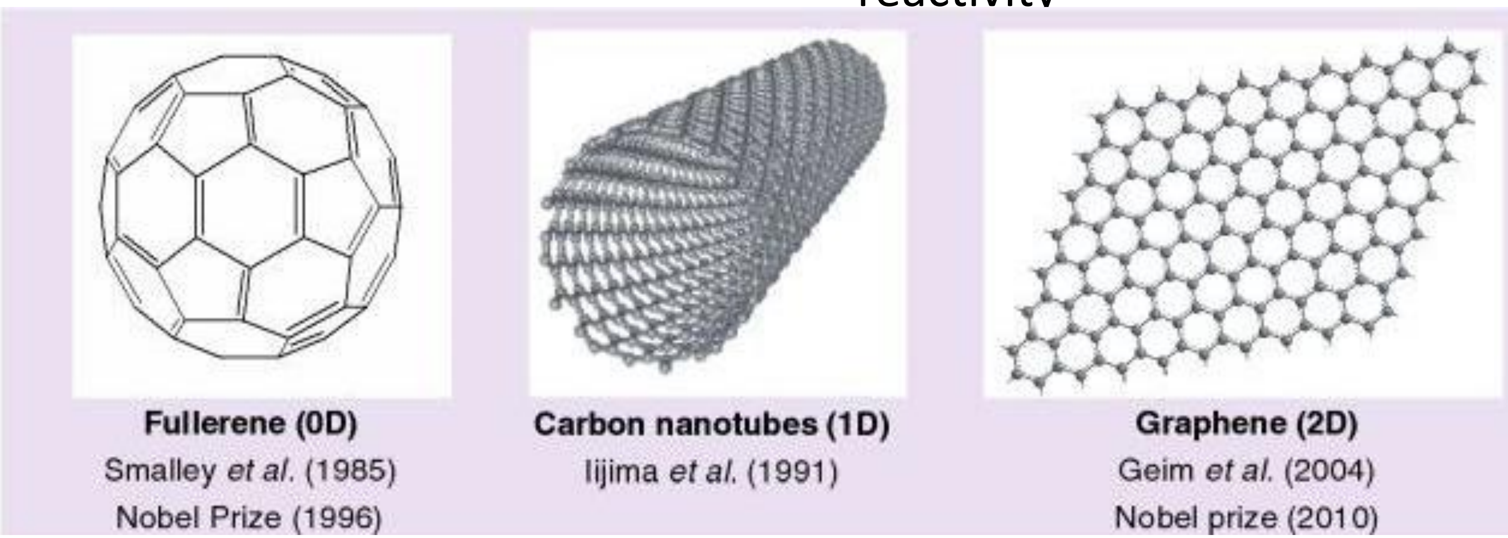
CNTs vs Graphene

Carbon Nanotubes (CNTs)

- Allotrope of carbon
- hollow, cylindrical structures, a sheet of graphene rolled into a cylinder.
- high thermal conductivity, electron mobility, and chemical reactivity

Graphene

- Allotrope of carbon
- 2D material, a single layer of graphite, with carbon atoms arranged in a hexagonal, honeycomb lattice.
- high thermal conductivity, electron mobility, and chemical reactivity



Base Powders

1

α-Si_3N_4 Powder

2

α-Si_3N_4 Powder

Oxidation at 1000 ˚ C for 10 hrs

3

α-Si_3N_4 Powder

Oxidation at 1000 ˚ C for 20 hrs

Materials Preparation

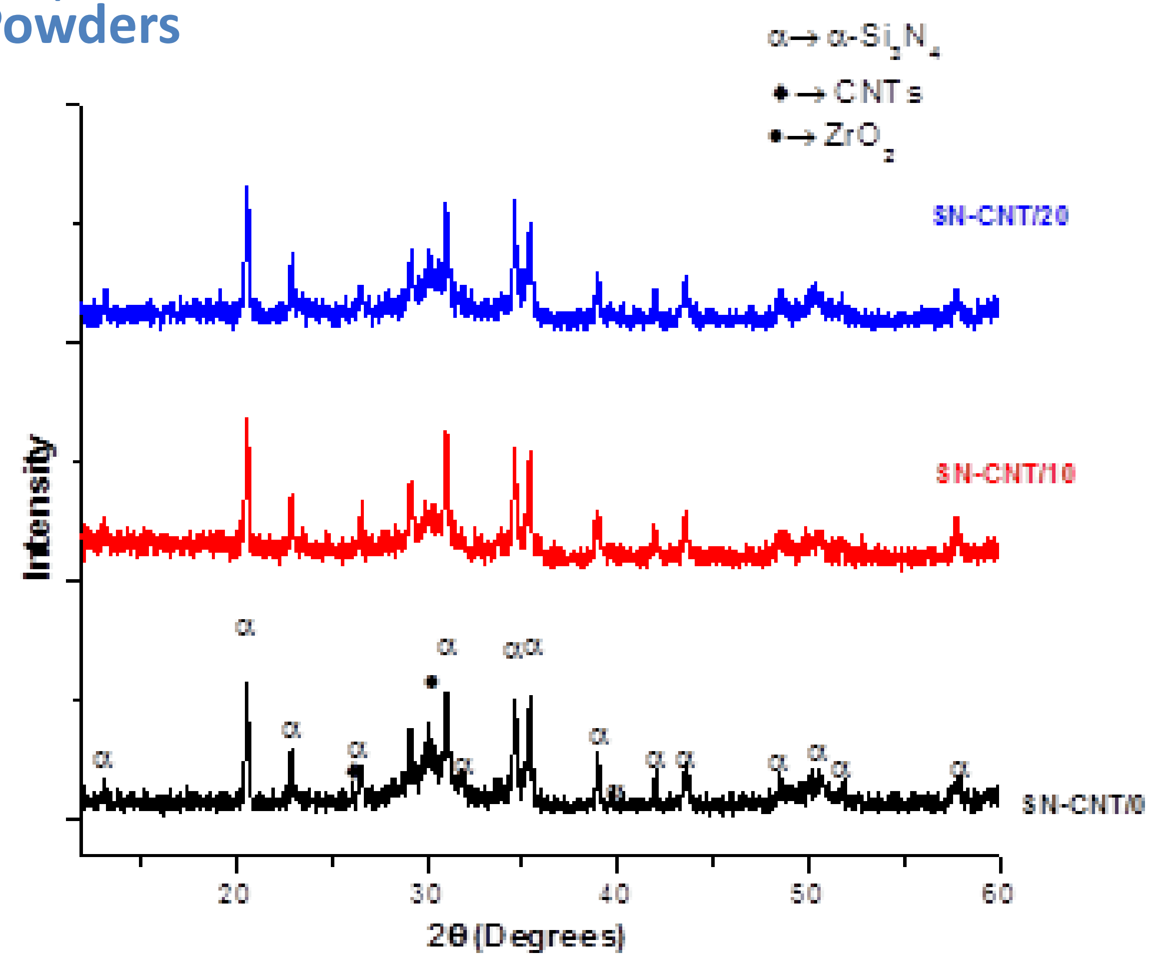
α-Si_3N_4 Powder → Mixing with 3 wt% MWCNTs → HIPing at 1700 ˚C for 3 hours

10 hrs oxidized α-Si_3N_4 → Mixing with 3 wt% MWCNTs → HIPing at 1700 ˚C for 3 hours

20 hrs oxidized α-Si_3N_4 → Mixing with 3 wt% MWCNTs → HIPing at 1700 ˚C for 3 hours

Characterization of samples

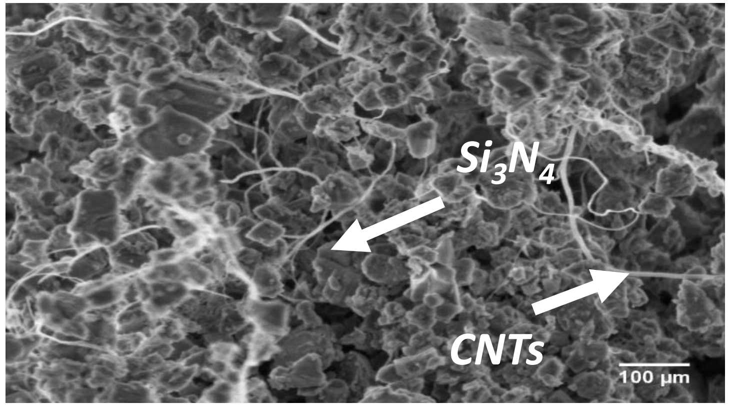
XRD of Si_3N_4-3% MWCNTs Powders

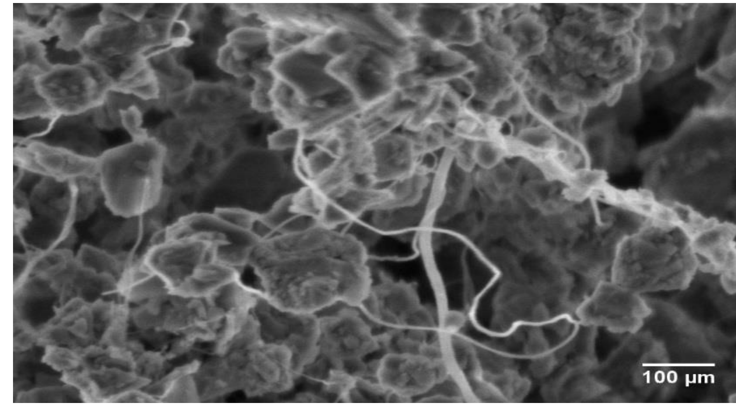


JCPDS PDF (01-076-1407), (00-33-1160), (00-47-1627) and (00-83-0944)

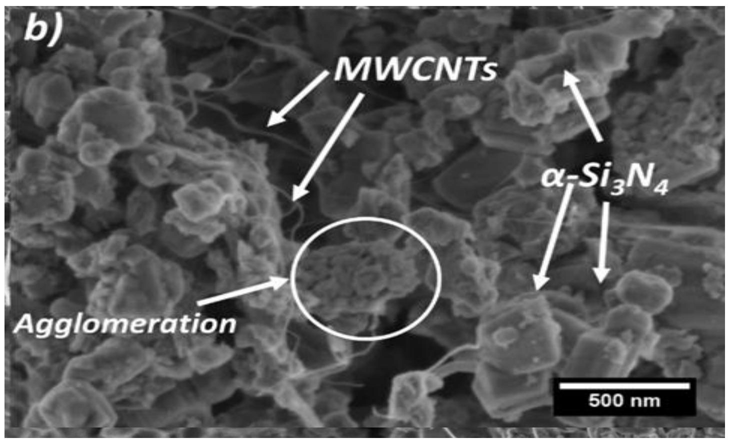
Morphological Study of Si_3N_4-3% MWCNTs

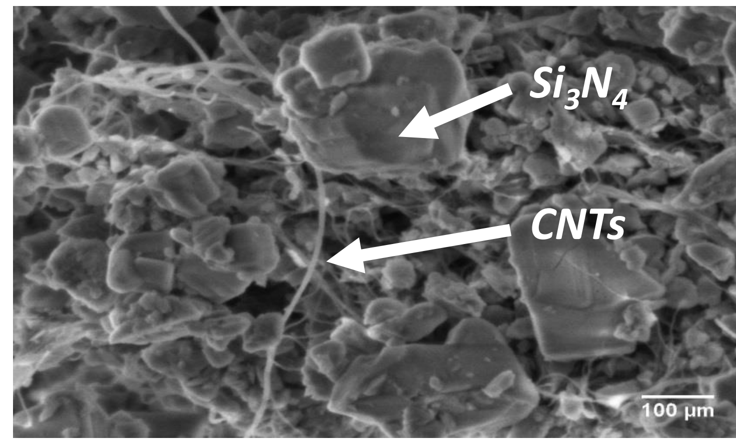
Unoxidized (Ref.)



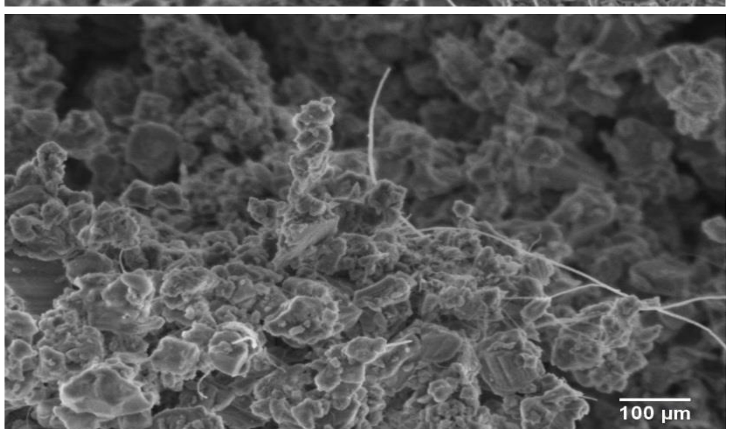


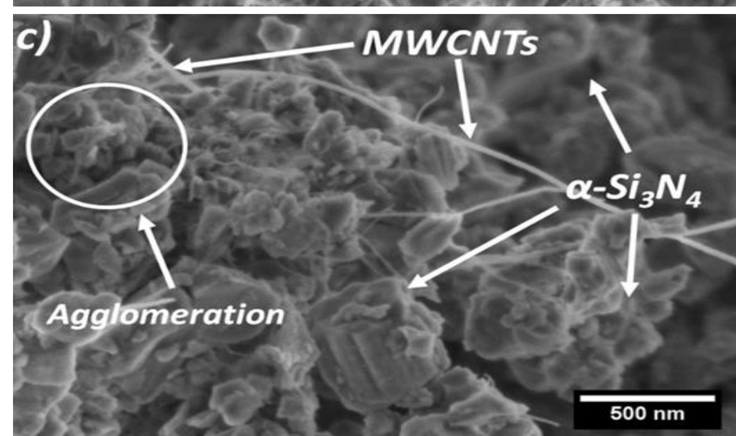
10 hrs oxidized



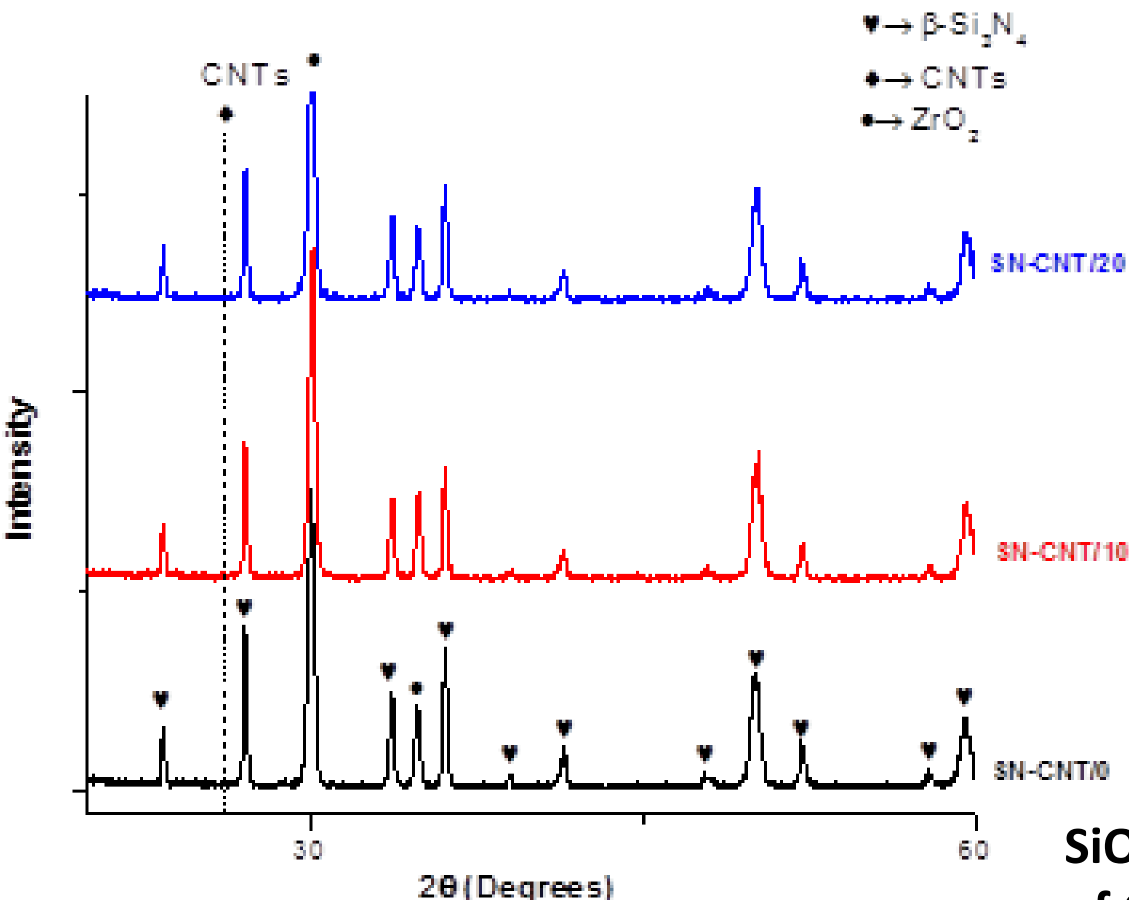


20 hrs oxidized





XRD after Sintering



~~$SiO_2 + Si_3N_4 = 2Si_2N_2O$~~

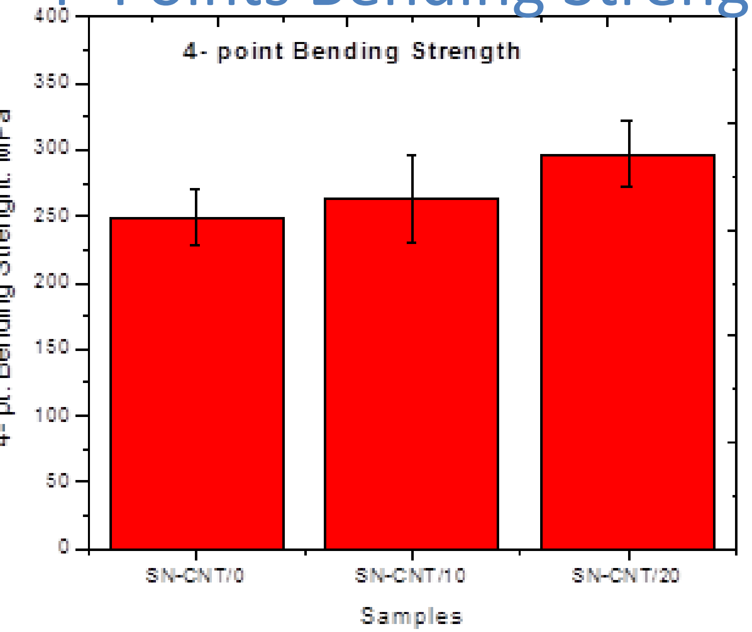
~~Si2N2O~~

SiO2 was consumed in the oxidation of CNTs

$$3SiO_2\,(l) + 6C + 2N_2 \leftrightarrow Si_3N_4 + 6CO$$

Flexural Strength

4- Points Bending Strength



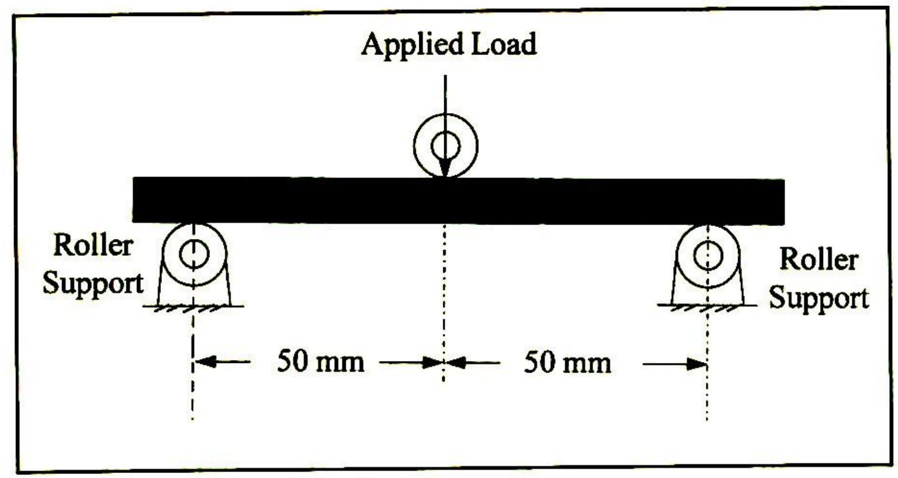
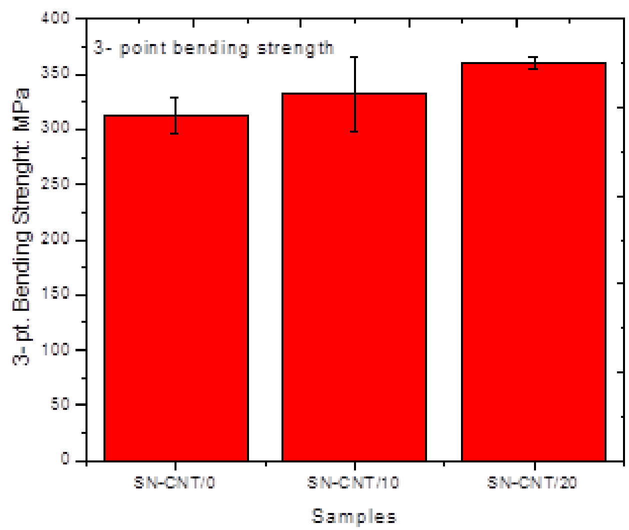
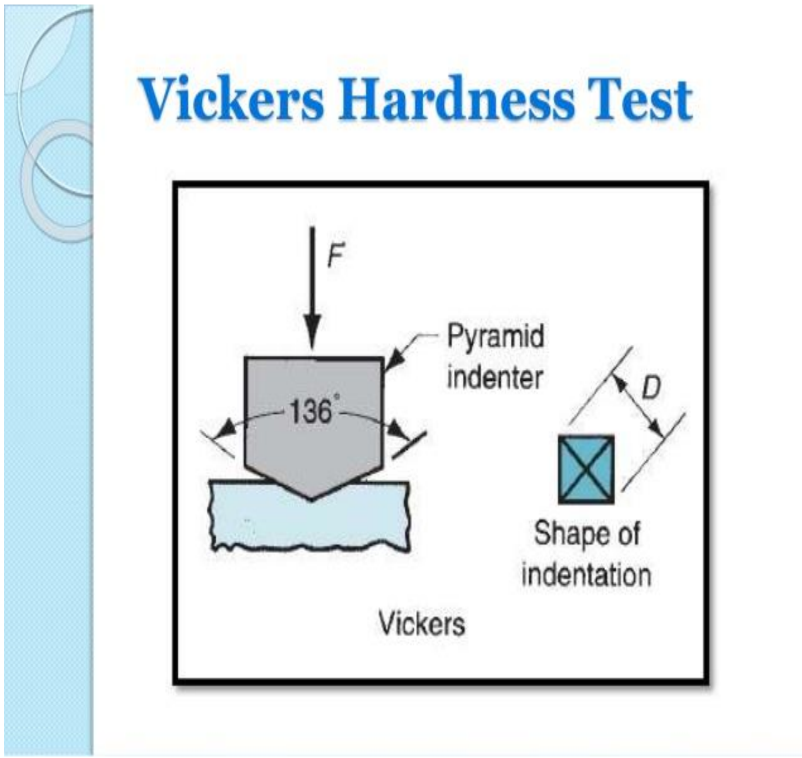


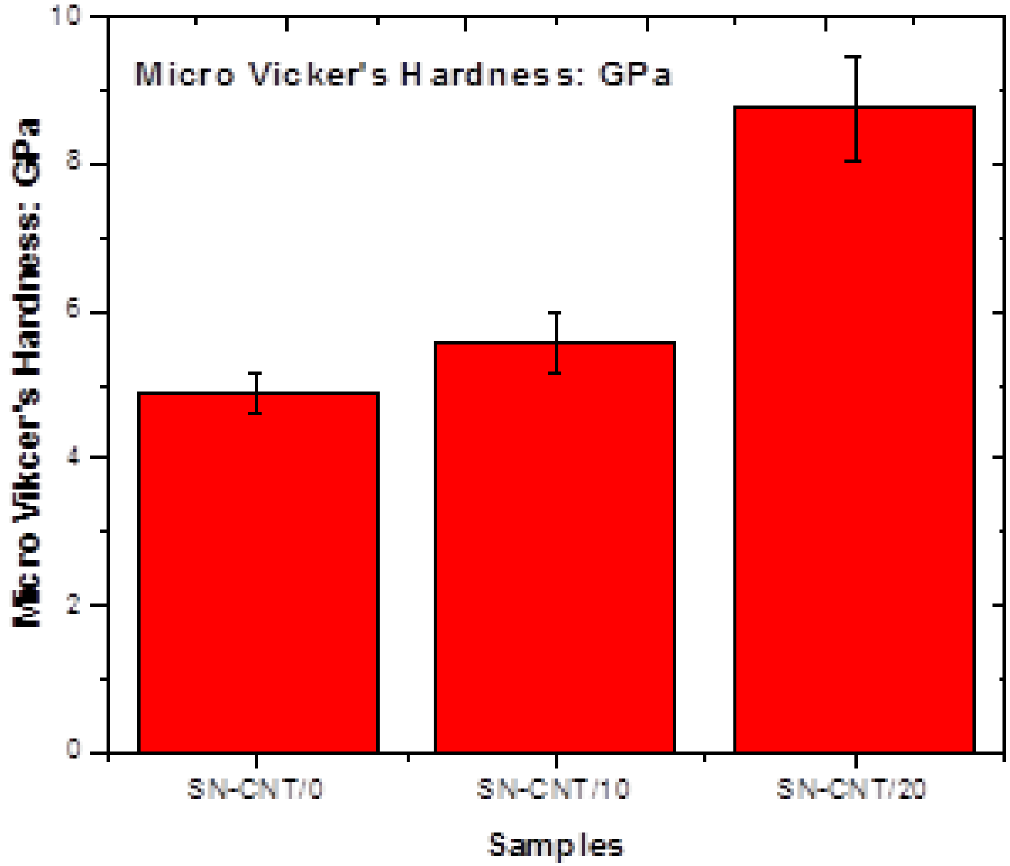
Fig 1: Schematic of three-point bending test.

3- Points Bending Strength



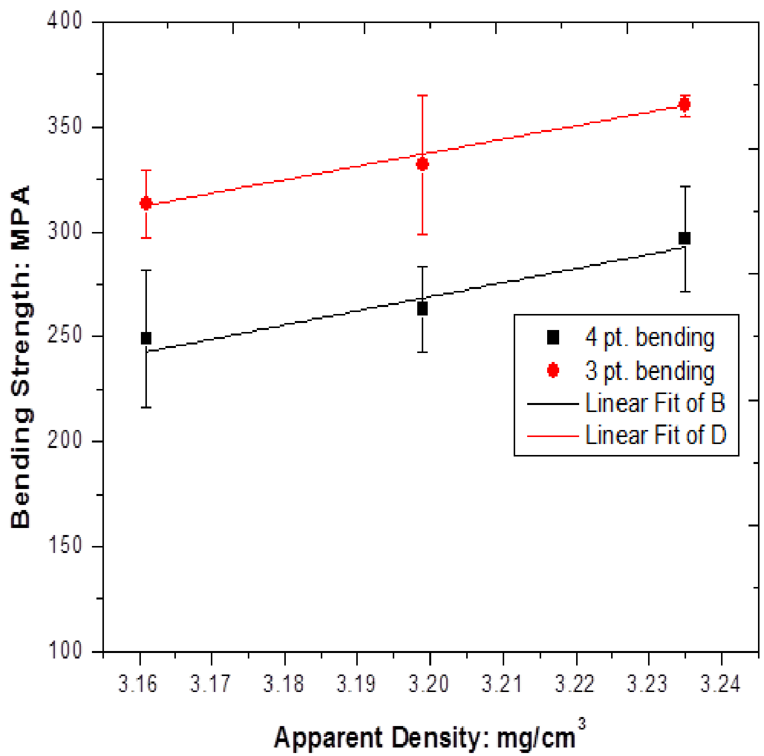
Vicker's Hardness

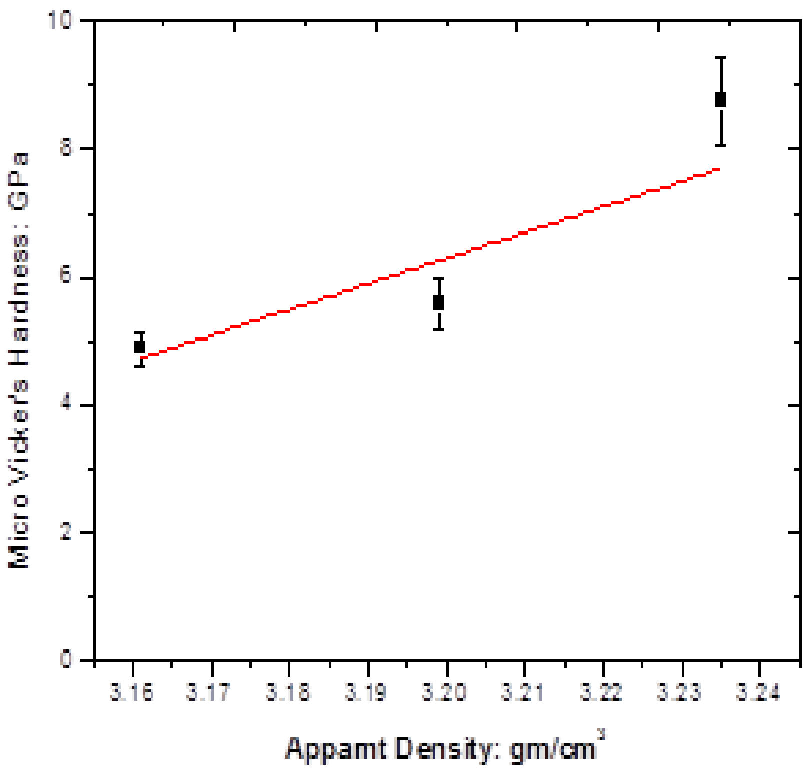




Mechanical Properties vs Density

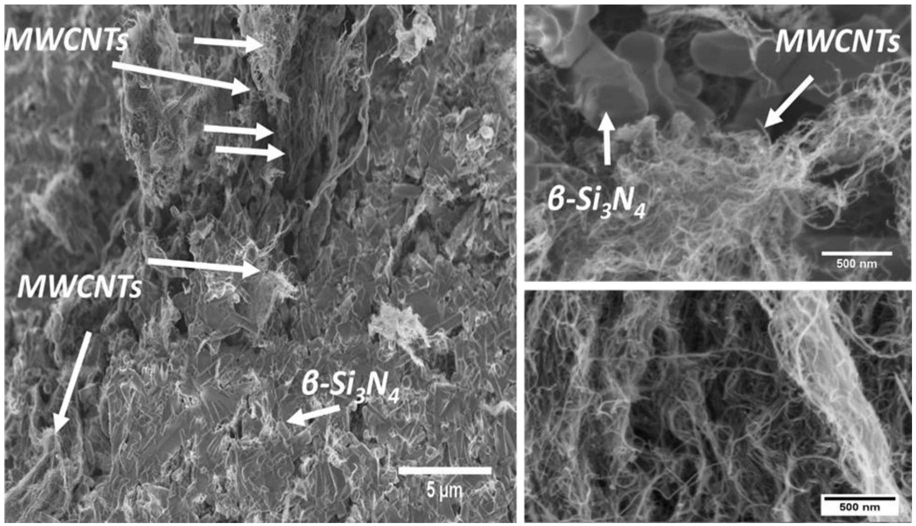
	SN-CNT/0	SN-CNT/10	SN-CNT/20
Apparent Density (gm/cm^3)	3.161	3.199	3.235
Relative Density (%)	97.71	98.88	100



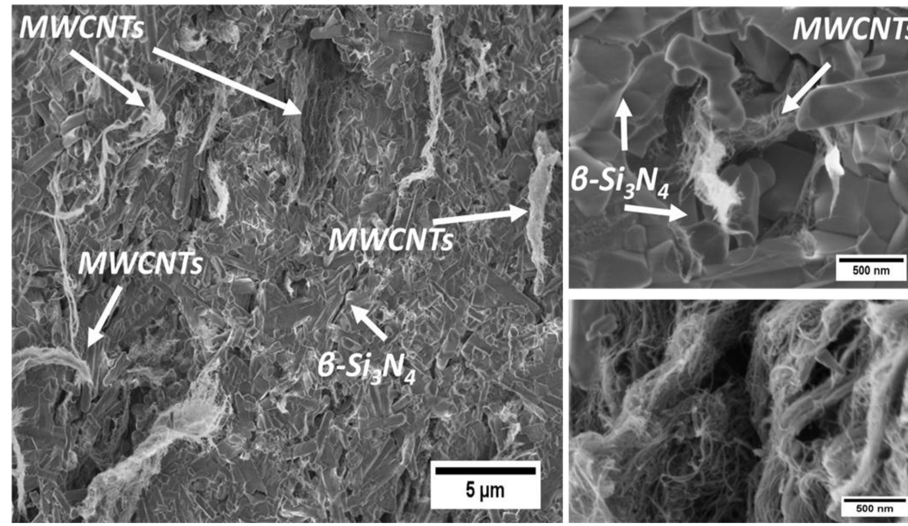


Fractured Surfaces

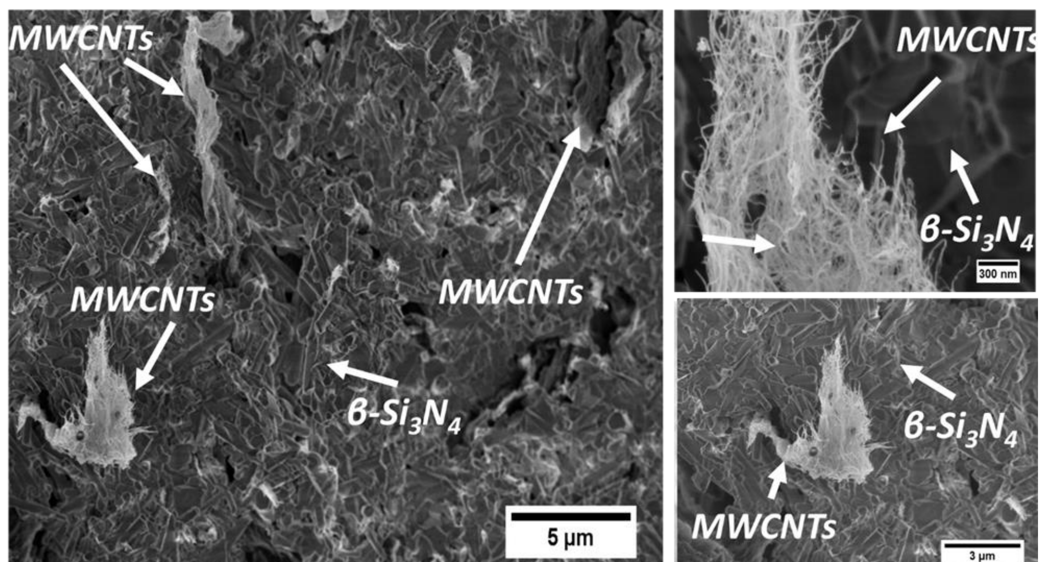
Reference materials



10 hrs oxidized



20 hrs oxidized



Conclusion

- The α – Si_3N_4 transformed to β – Si_3N_4 completely at 1700 ˚C during sintering.
- The oxidation of starting powder caused the increase in apparently density of sintered samples which was beneficial for the mechanical properties of the composite.
- The CNTs were found in clusters form between the intergranular spaces of β- Si_3N_4 matrix.
- Neither the Si_2N_2O was found and nor the structural change before and after the sintering.
- The strength and hardness of the material was the function of apparent density.
- Higher apparent density and higher the hardness and flexural strength was observed.

Future Plans

- Preparation of 3rd article for the Journal with Impact Factor on Si_3N_4 with the addition of Graphene and CNTs.
- Study the mechanism of formation of CO2 and CO during the sintering process.
- Exams and Complex Exams

Future Plans for Conferences

- Junior EURO-MAT 2018 (Abstract Accepted).

Acknowledgement

- Dr. Zsolt Fogarassy for TEM & HRTEM and Dr. Zsolt E. Horvath for XRD.
- Thanks to supervisors for their support.
- Special Thanks to the Technical Staff Viktor Varga, Sandor Gurban; for their support.

Thank you for your attention