

Si_3N_4 Ceramic Composite with the addition of MWCNTs and ML-Graphene

Awais Qadir^{a,b}

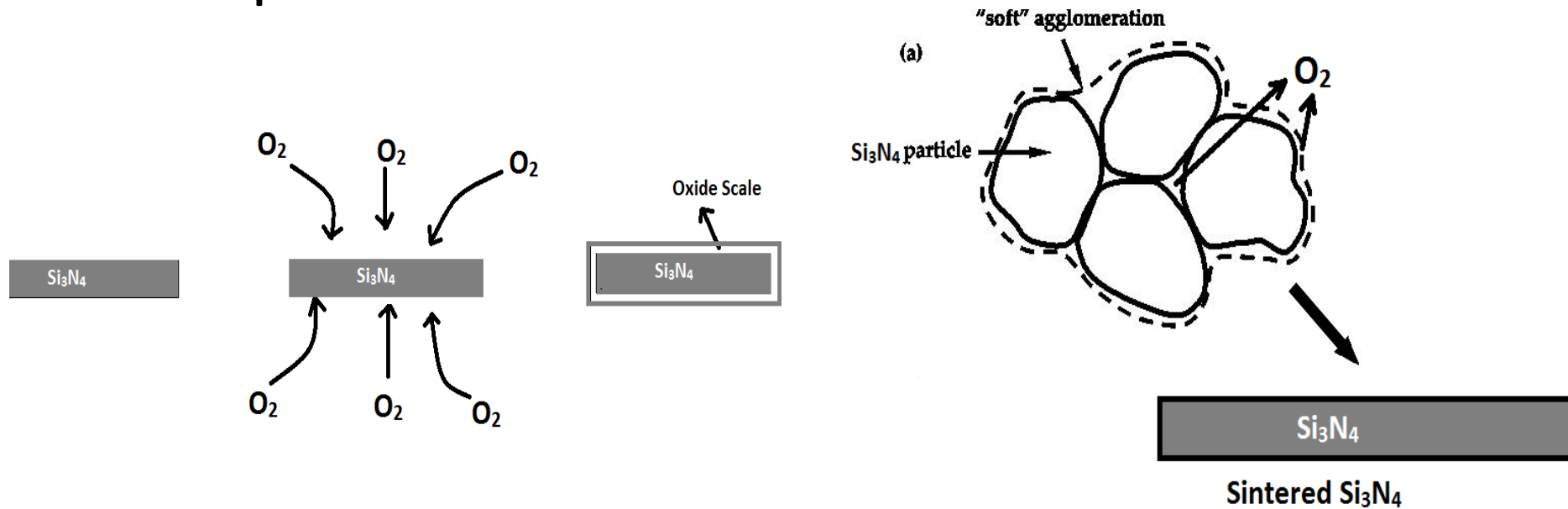
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Aim of work

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



Plans Accomplished

1st Semester

Si ₃ N ₄	
1434	Si ₃ N ₄ (Ref.)
1435	Si ₃ N ₄ (10 hrs Oxidized)
1436	Si ₃ N ₄ (20 hrs Oxidized)

2nd Semester

Addition of 1% CNTs and 2% Graphene	
1459	Si ₃ N ₄ - 1% CNTs – 2% Graphene (Ref.)
1460	Si ₃ N ₄ (10 hrs oxidized) – 1% CNTs – 2% Graphene
1461	Si ₃ N ₄ (20 hrs oxidized) – 1% CNTs – 2% Graphene
Addition of 3% CNTs	
1462	Si ₃ N ₄ – 3% CNTs (Ref.)
1463	Si ₃ N ₄ (10 hrs oxidized) – 3% CNTs
1464	Si ₃ N ₄ (20 hrs Oxidized) – 3% CNTs
Addition of 3 wt.% Graphene	
1468	Si ₃ N ₄ – 3% Graphene (Ref.)
1469	Si ₃ N ₄ (10 hrs Oxidized) – 3% Graphene
1470	Si ₃ N ₄ (20 hrs oxidized) – 3% Graphene

Progress Report 2017-2018

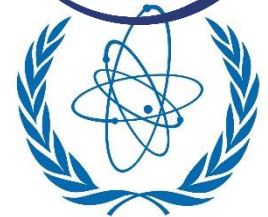
- Paper has been submitted in the **Journal of European Ceramic Society** (Impact Factor 3.411) (Submitted)



Participation in Conferences 2017-2018



- 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 Theoretical 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](#)



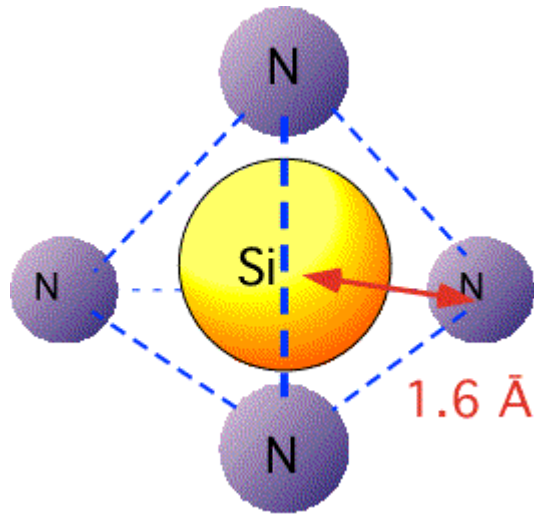
IAEA

International Atomic Energy Agency



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.



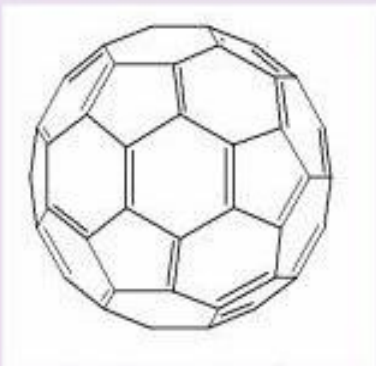
CNTs vs Graphene

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



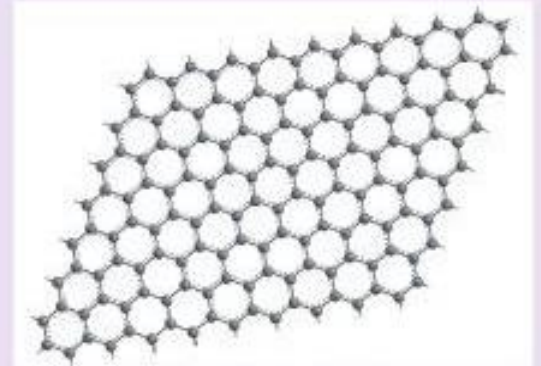
Fullerene (0D)

Smalley *et al.* (1985)
Nobel Prize (1996)



Carbon nanotubes (1D)

Iijima *et al.* (1991)



Graphene (2D)

Geim *et al.* (2004)
Nobel prize (2010)

Base Powders

1

α -Si₃N₄
Powder

2

α -Si₃N₄
Powder

3

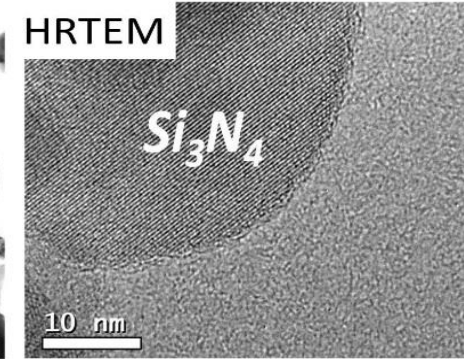
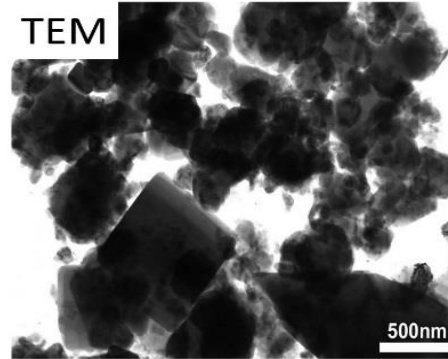
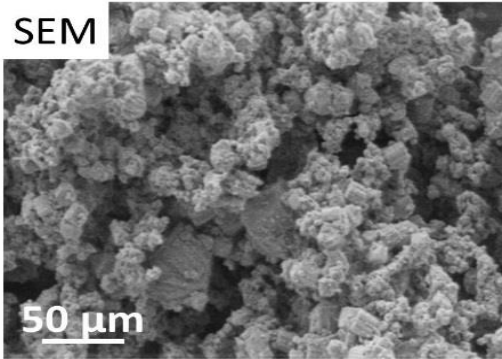
α -Si₃N₄
Powder

Oxidation
at 1000 ° C
for 10 hrs

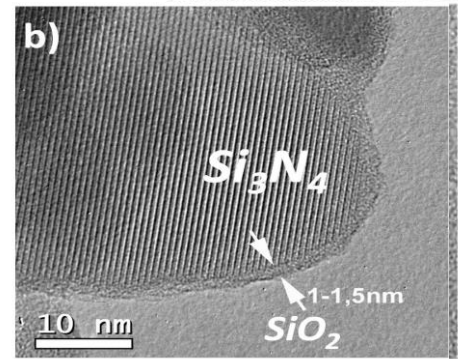
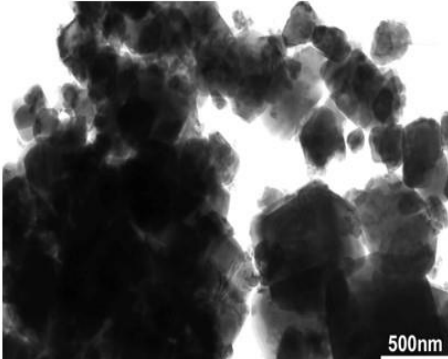
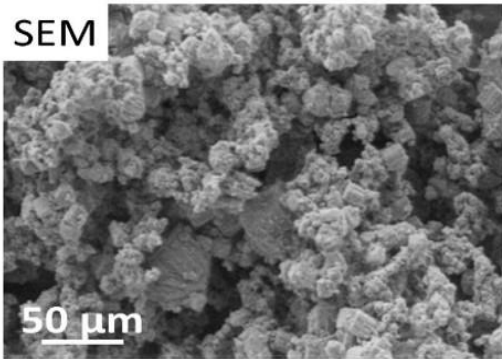
Oxidation
at 1000 ° C
for 20 hrs

Structure of Base powders

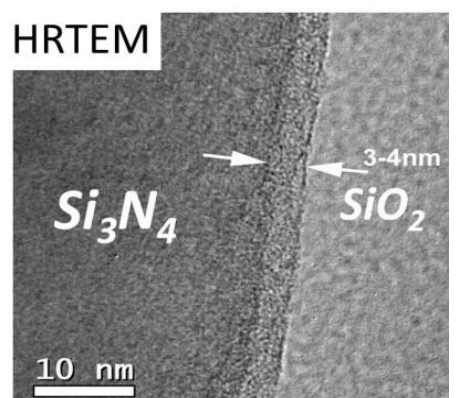
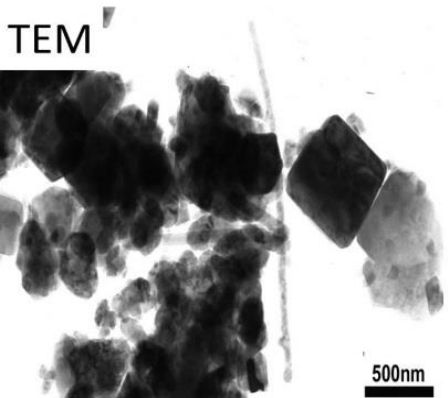
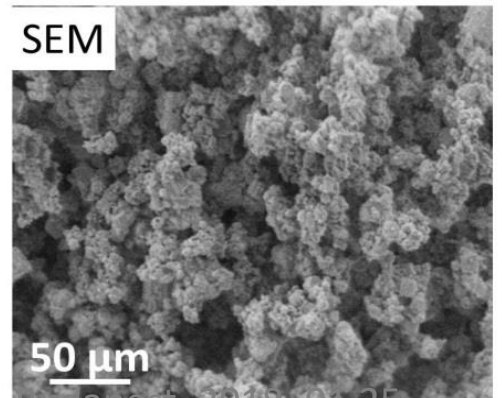
α - Si_3N_4
Powder



10 hrs
oxidized
 α - Si_3N_4

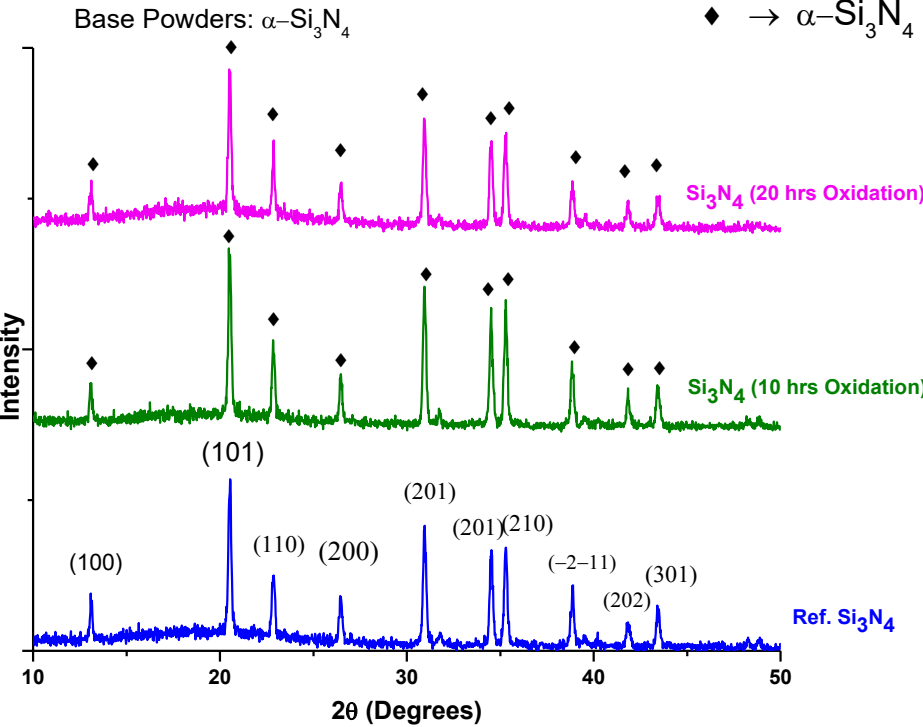


20 hrs
oxidized
 α - Si_3N_4

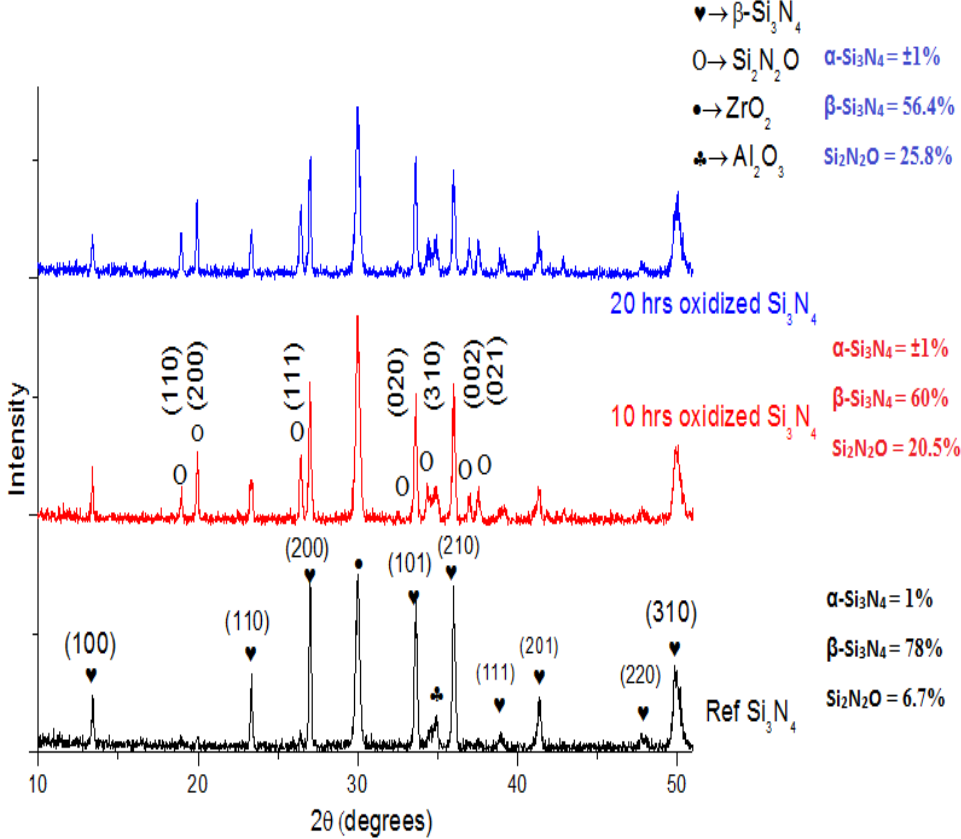


Characterization of powder and sintered sample

Base Powder



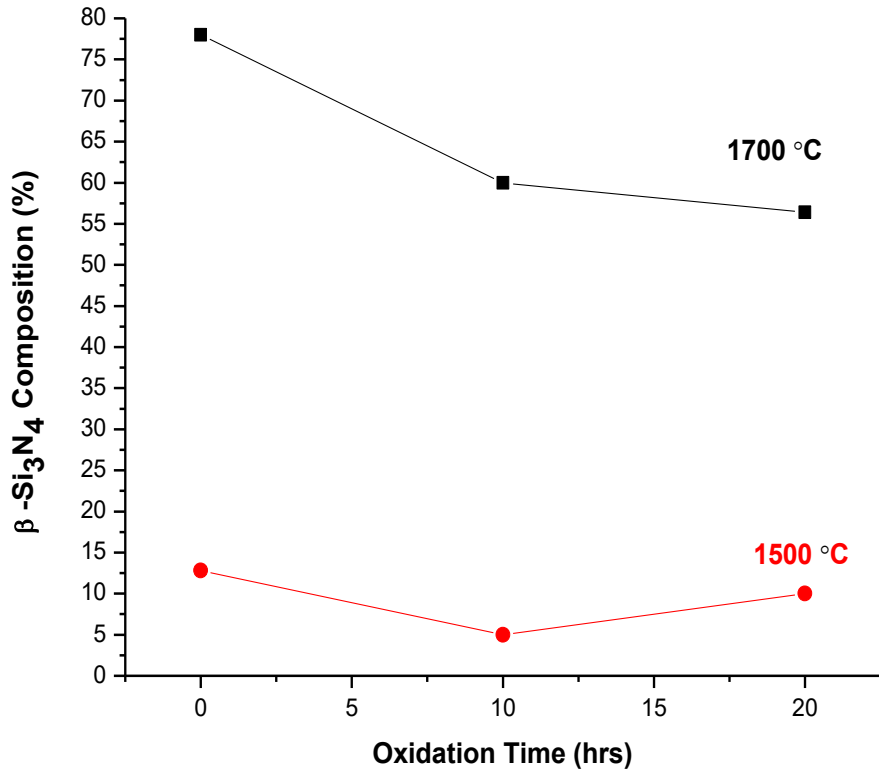
Sintered at 1700 °C



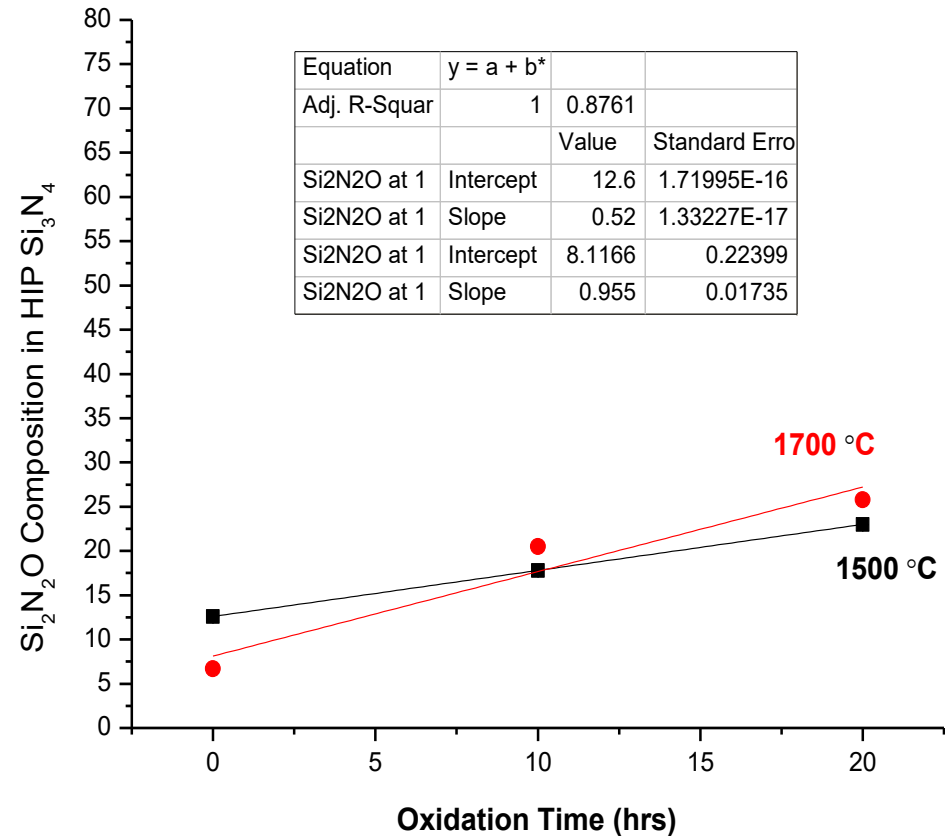
JCPDS PDF (01-076-1407), (00-33-1160), (00-47-1627) and (00-83-0944)

Characterization Sintered Samples

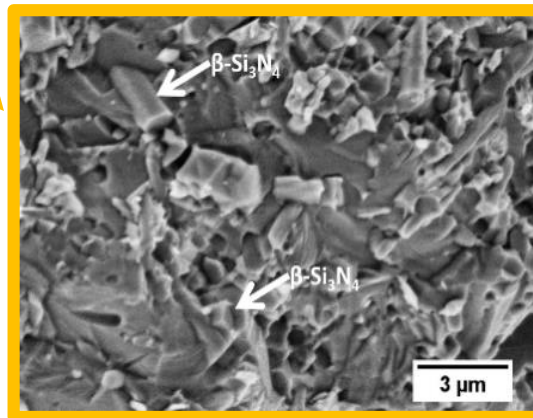
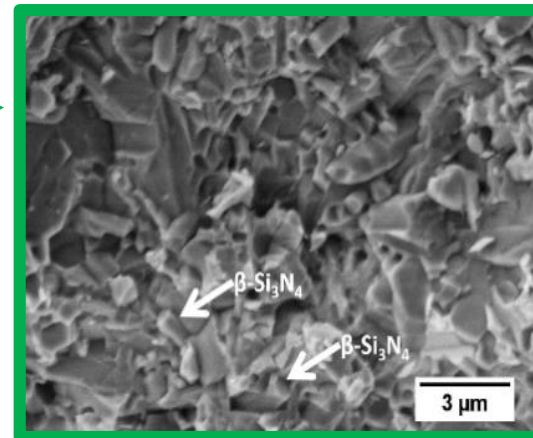
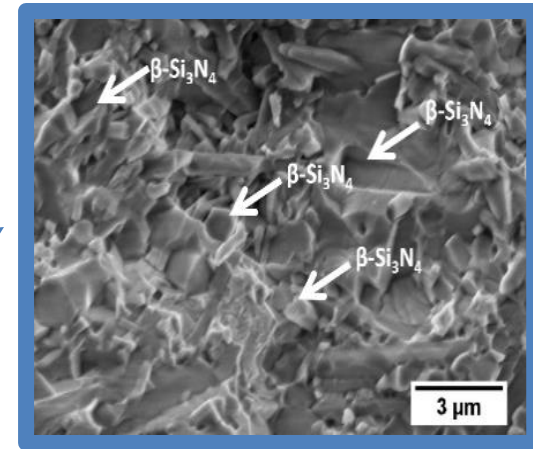
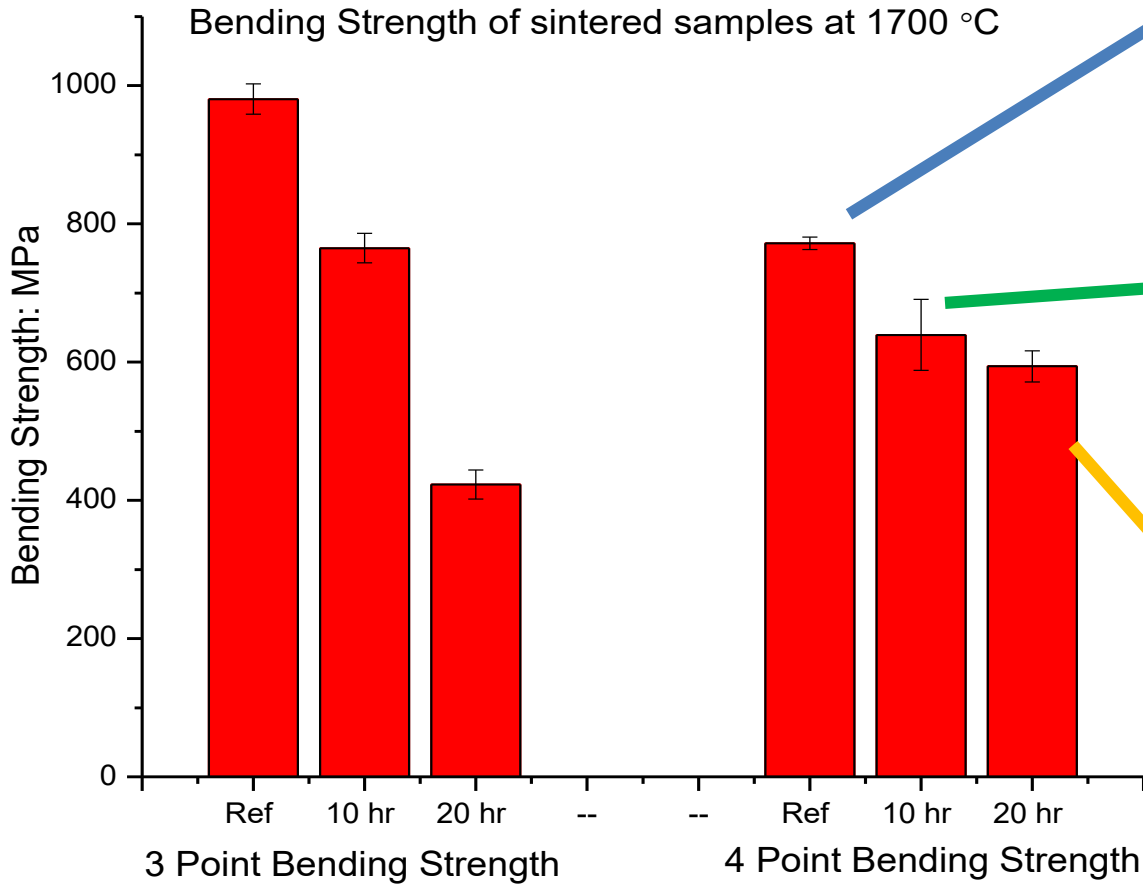
β -Si₃N₄ Content in HIP Si₃N₄



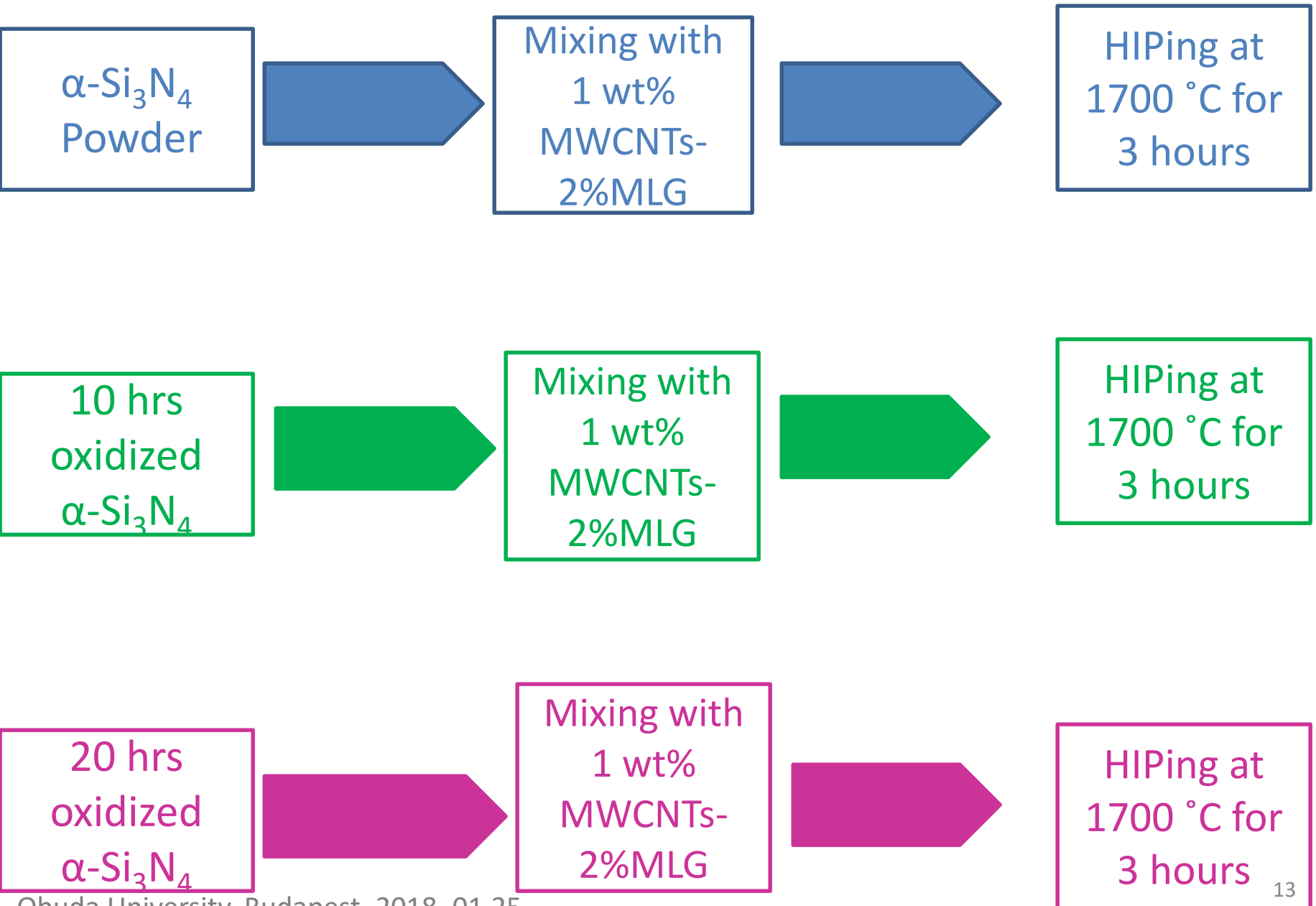
Si₂N₂O Content in HIP Si₃N₄



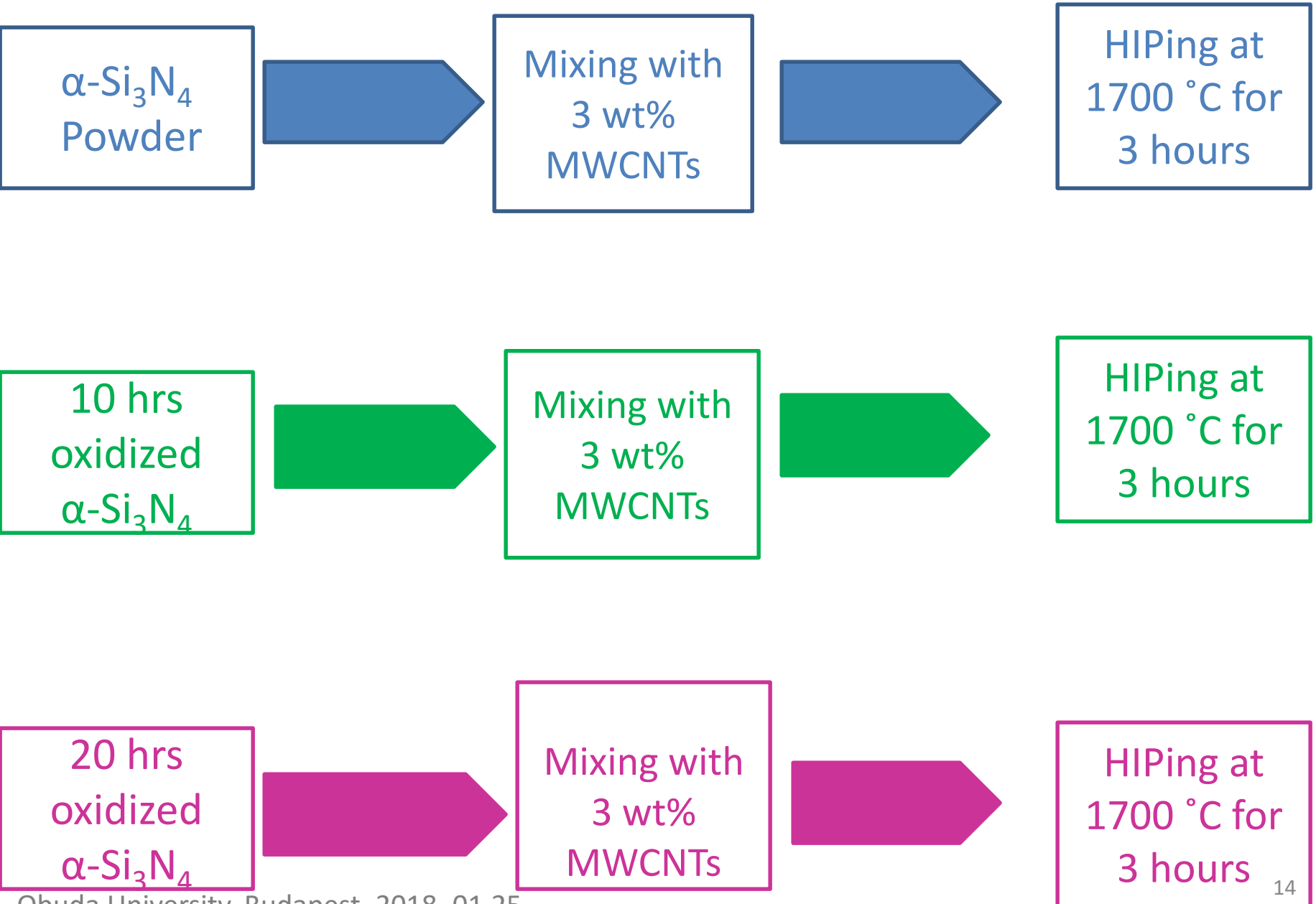
Bending Strength



Materials Preparation

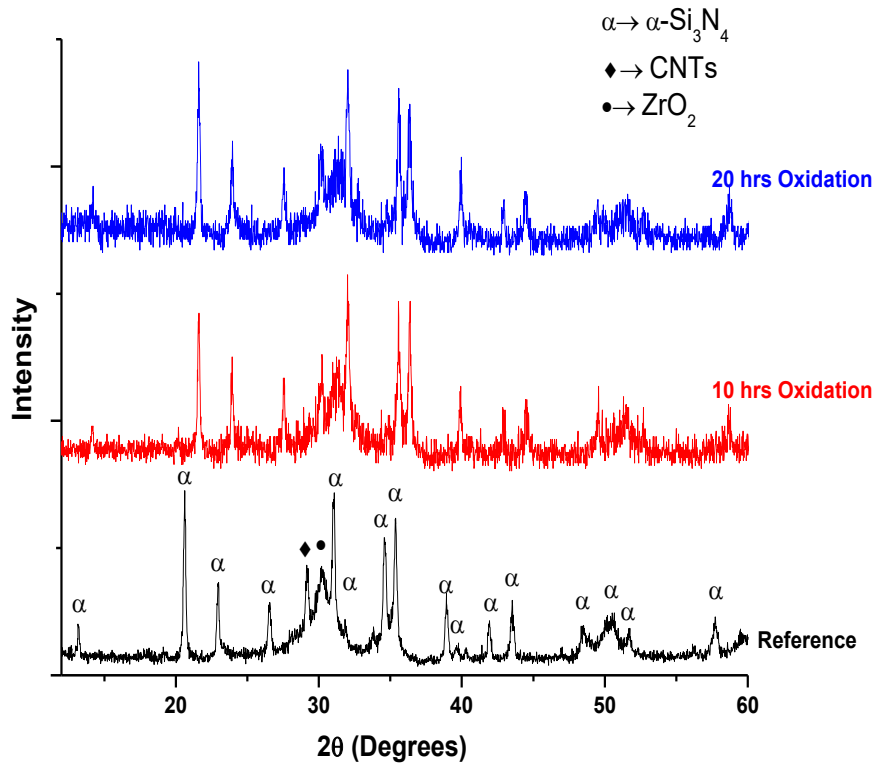


Materials Preparation

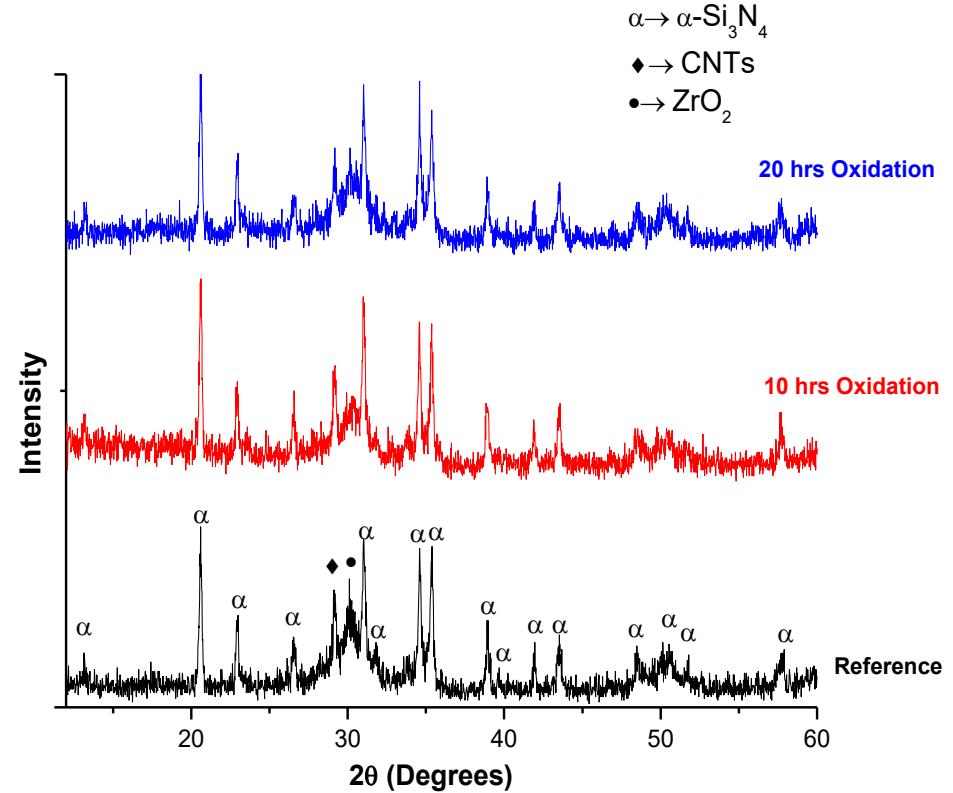


Characterization of samples

Si₃N₄-1% MWCNTs-2%MLG



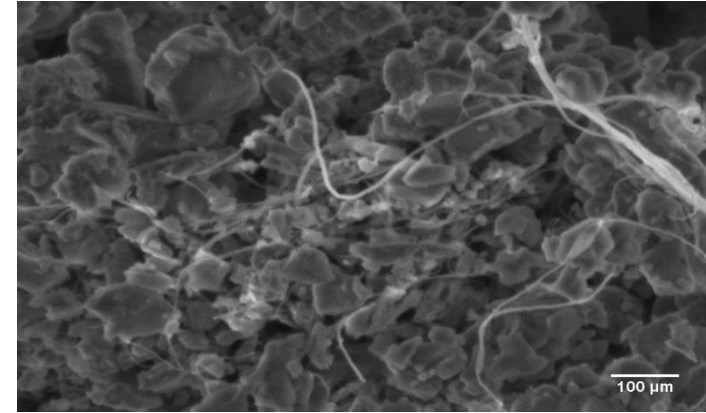
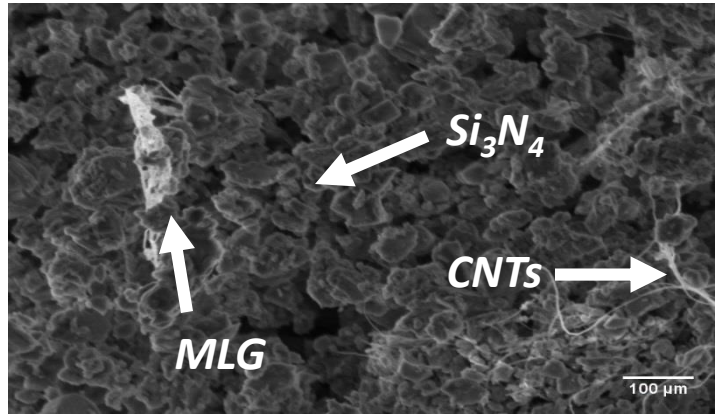
Si₃N₄-3% MWCNTs



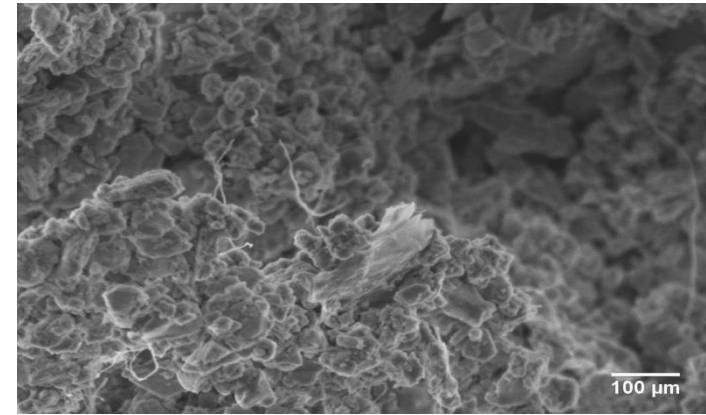
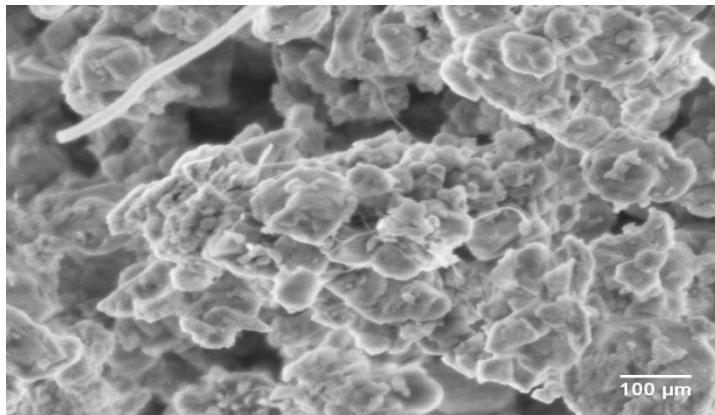
JCPDS PDF (01-076-1407), (00-33-1160), (00-47-1627) and (00-83-0944)

Morphological Study of Si_3N_4 -1%MWCNTs-2%MLG

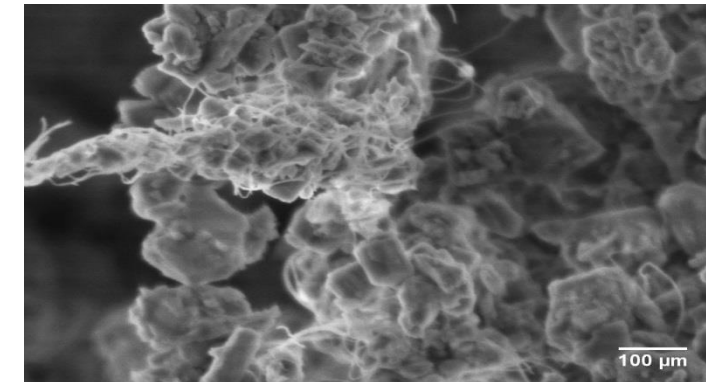
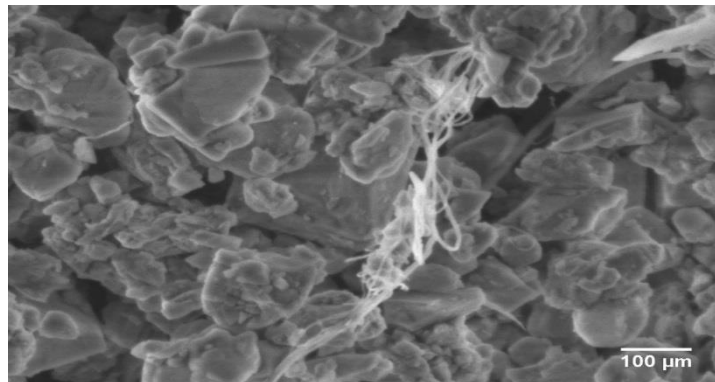
Unoxidized
(Ref.)



10 hrs
oxidized

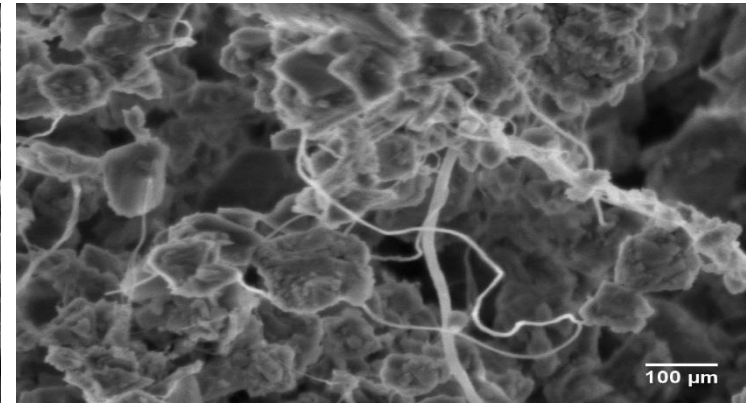
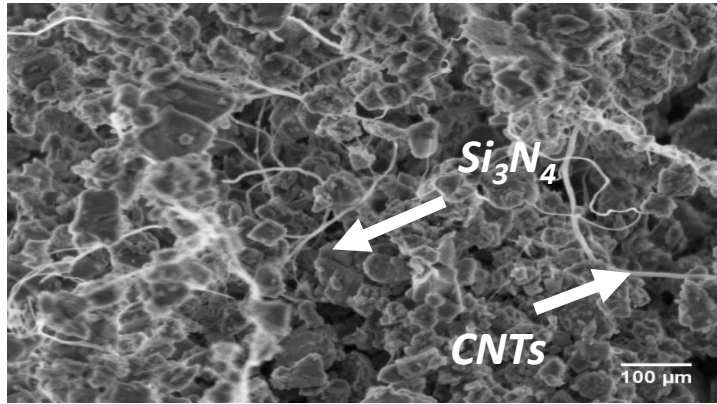


20 hrs
oxidized

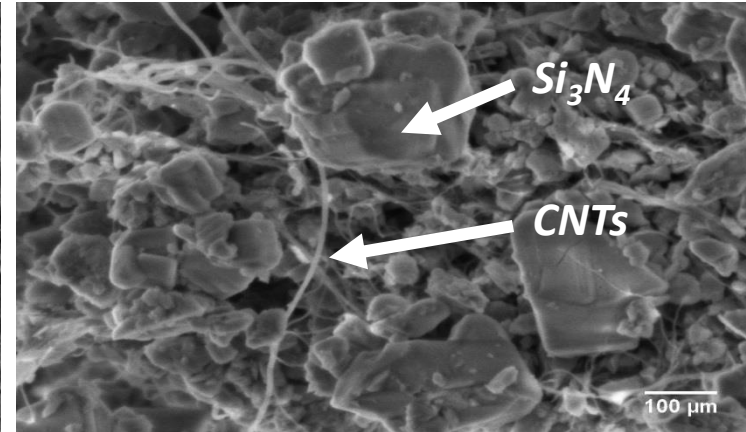
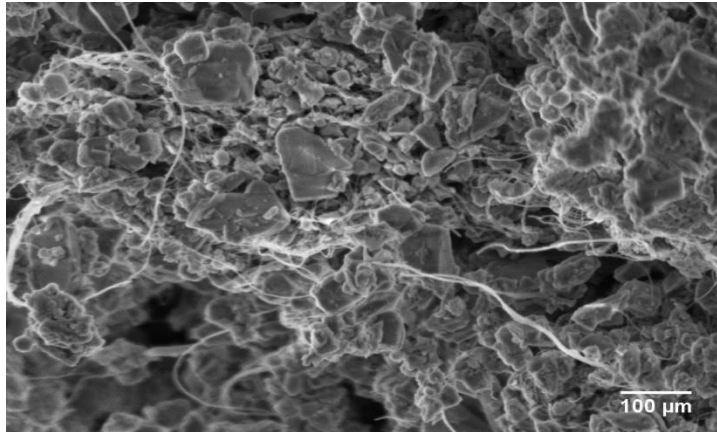


Morphological Study of Si_3N_4 -3% MWCNTs

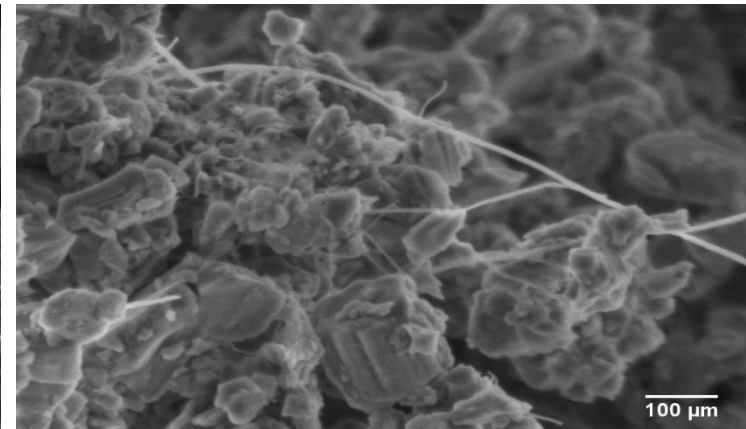
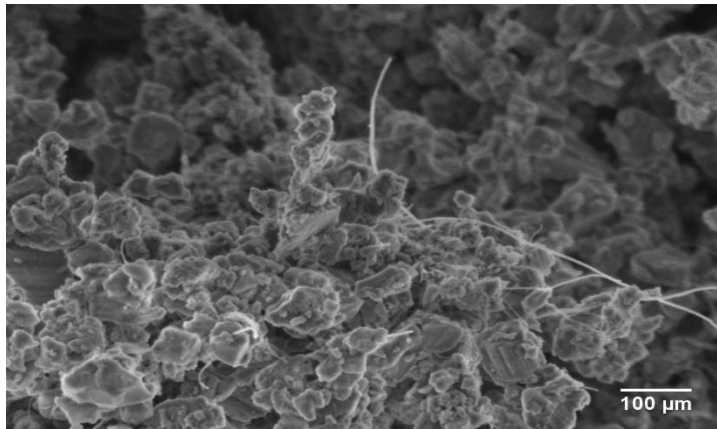
Unoxidized
(Ref.)



10 hrs
oxidized



20 hrs
oxidized



Conclusion

- Powders were milled successfully by attrition milling
- Particles size in between 50 nm to 500 nm
- Oxidation of base powder was done successfully and confirmed by EDX, HRTEM.
- MWCNTs and MLG mixed with the base powder successfully without its deformation.
- Uniform distribution of MWCNTs and Graphene in the powder.
- Further investigation are needed for the CNTs interaction with the matrix (Si_3N_4).

Future Plans

- Sintering by HIP
- Characterization of Sintered Samples by TEM, HRTEM, SEM, EDX.
- Mechanical Testing (HV, 3 pt. & 4pt. Bending st., toughness, Young's Modulus and surface wear testing)
- Paper Publication in International Journal

Future Plans for Conferences

- Junior EURO-MAT 2018 (Abstract Submitted).

FEMS Junior EUROMAT 2018

The Main Event for Young Materials Scientists

July 8-12, 2018 / Budapest, Hungary

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 and Sandor Gurban

Thank you for your attention

