Óbudai Egyetem

Silicon Nitride composites with the addition of CNTs – A review

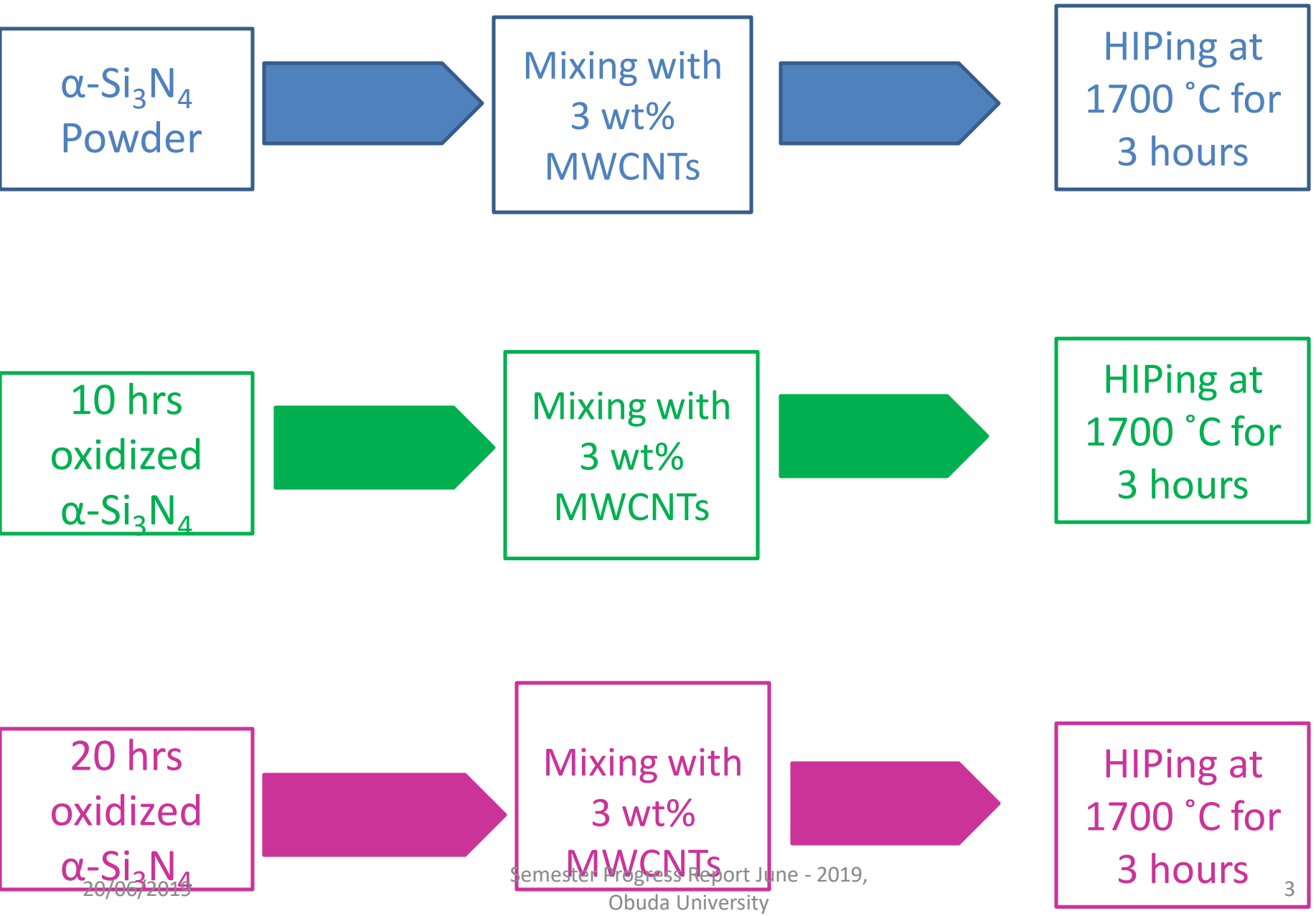
Tribological Properties of Si_3N_4+3 wt. % MWCNTs

Awais Qadir

Supervisor: Prof. Dr. Dusza János

Tribological Properties of Si_3N_4+3 wt. % MWCNTs

Materials Preparation

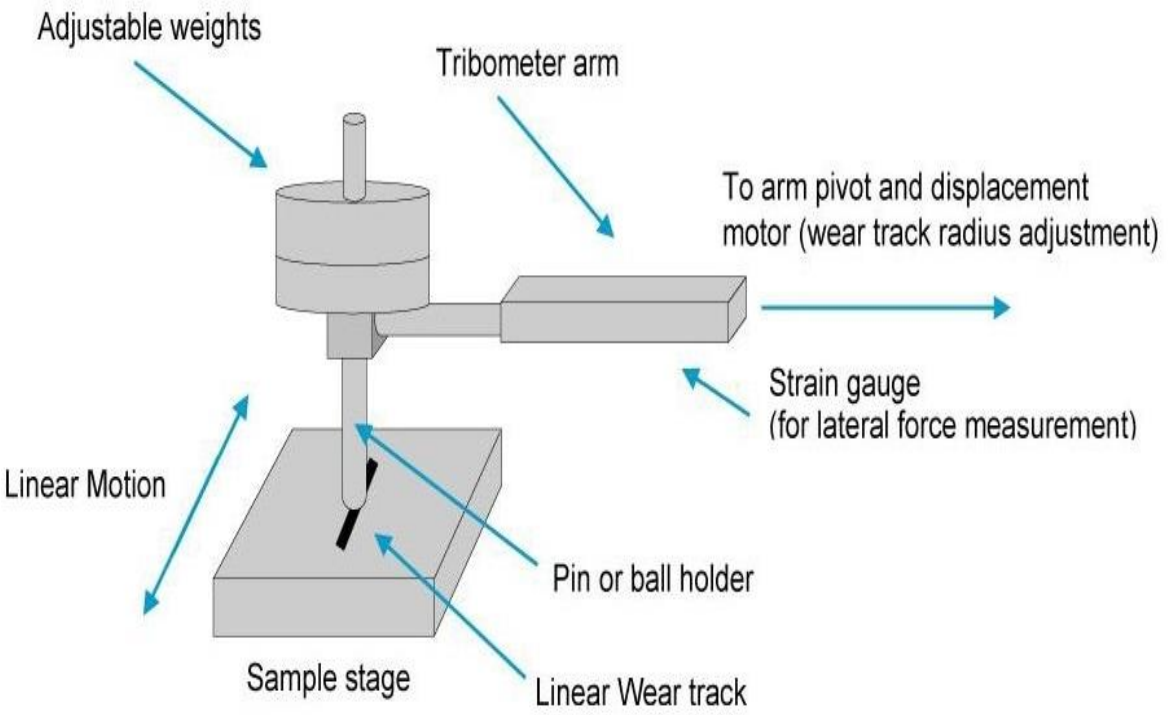


Density of composites

	Un-oxidized S_3N_4-3 wt. % MWCNT	10 hours oxidized S_3N_4-3 wt. % MWCNT	20 hours oxidized S_3N_4-3 wt. % MWCNT
Apparent Density (gm/cm^3)	3.161	3.199	3.235
Relative Density (%)	93.4	94.5	95.6

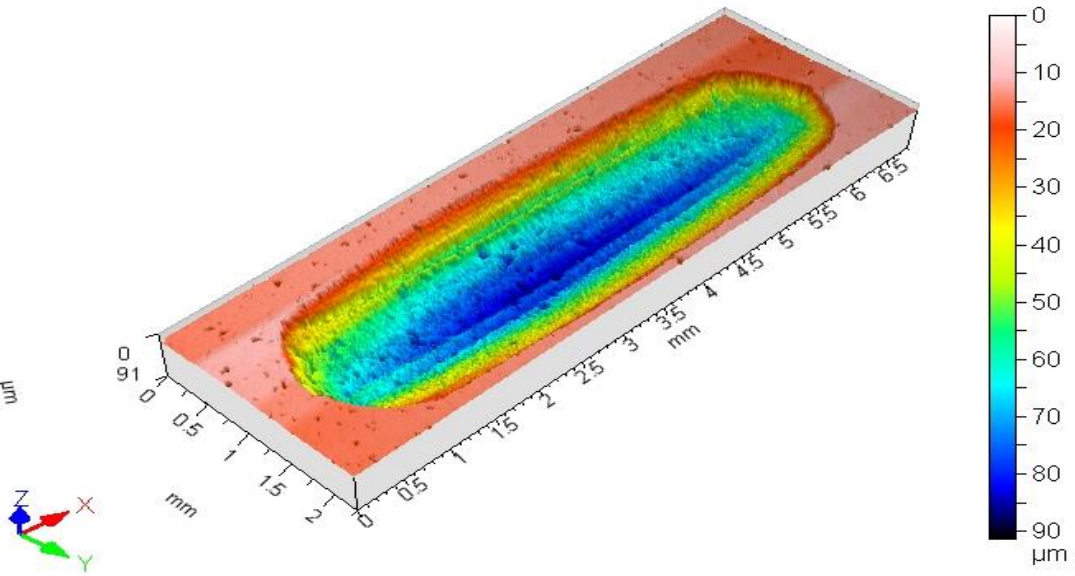
Tribological Testing

Resprocatory ball-on-plate Technique,
Applied load 13.5 N, Sliding speed 10 mm/s, Sliding distance 720 m, Dry conditions, Room temperature, Ball Si_3N_4

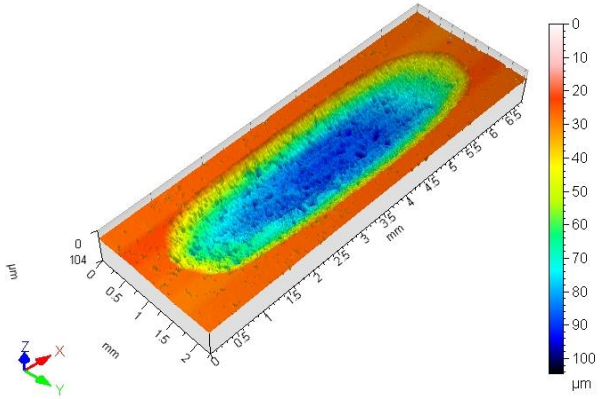


Confocal Microscopic image of Wear Track

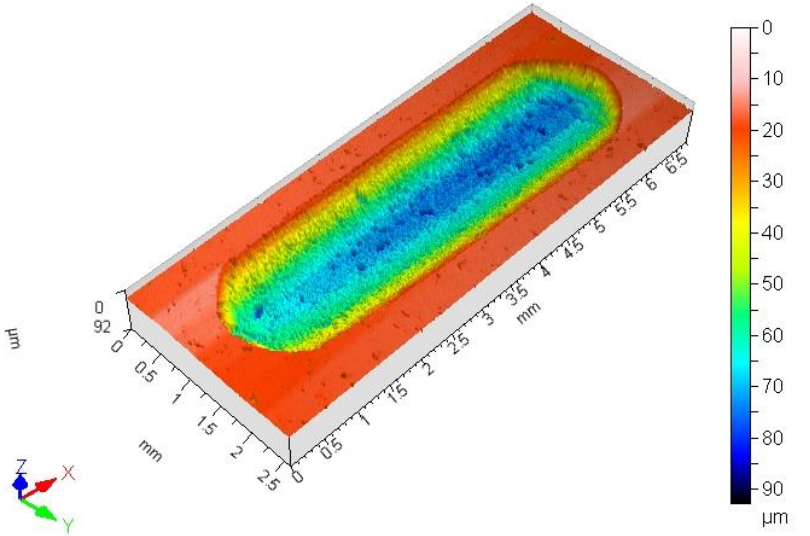
Un-oxidized Si_3N_4 + 3 wt. % MWCNTs



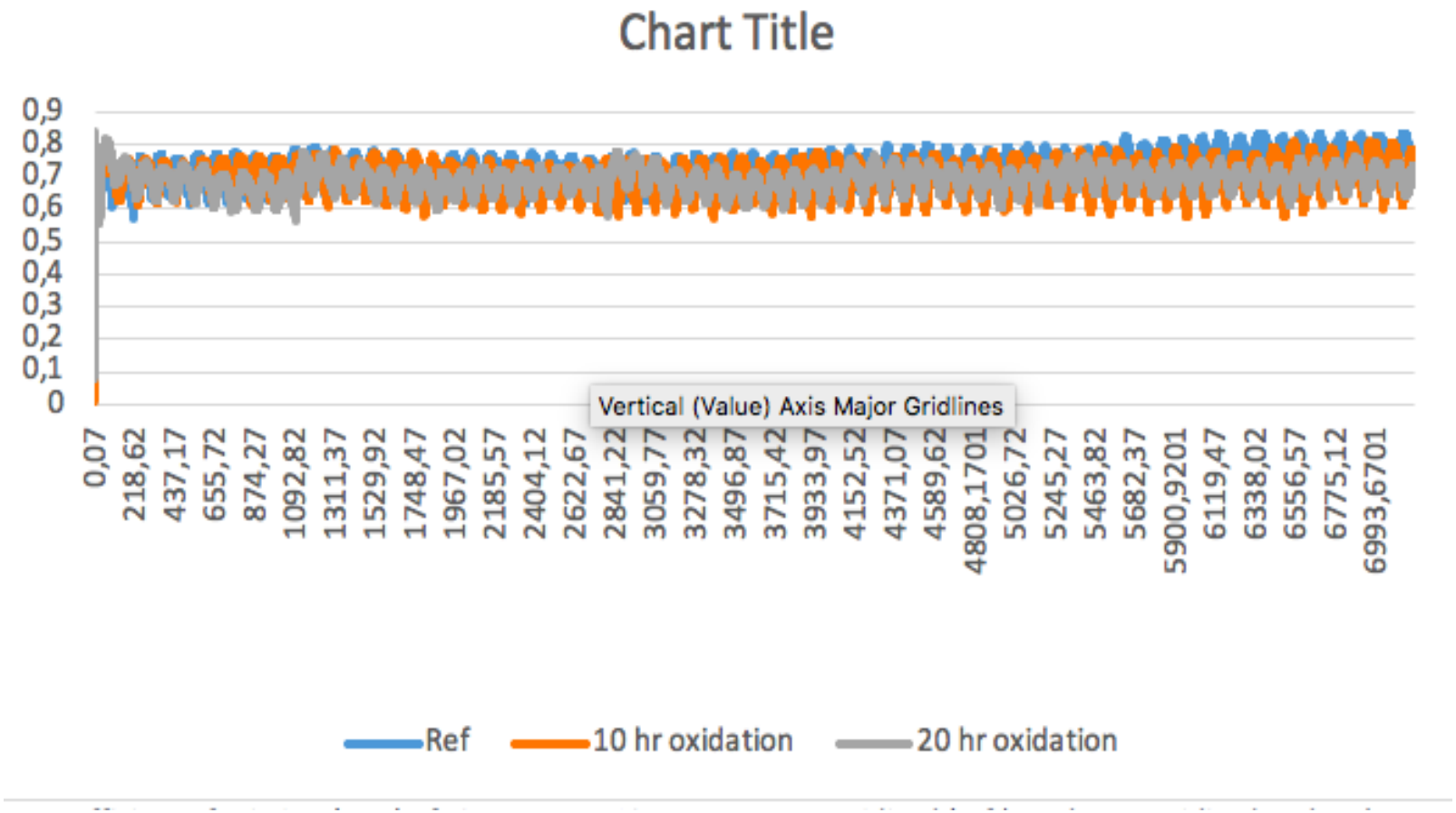
10 hrs oxidized Si_3N_4 + 3 wt. % MWCNTs



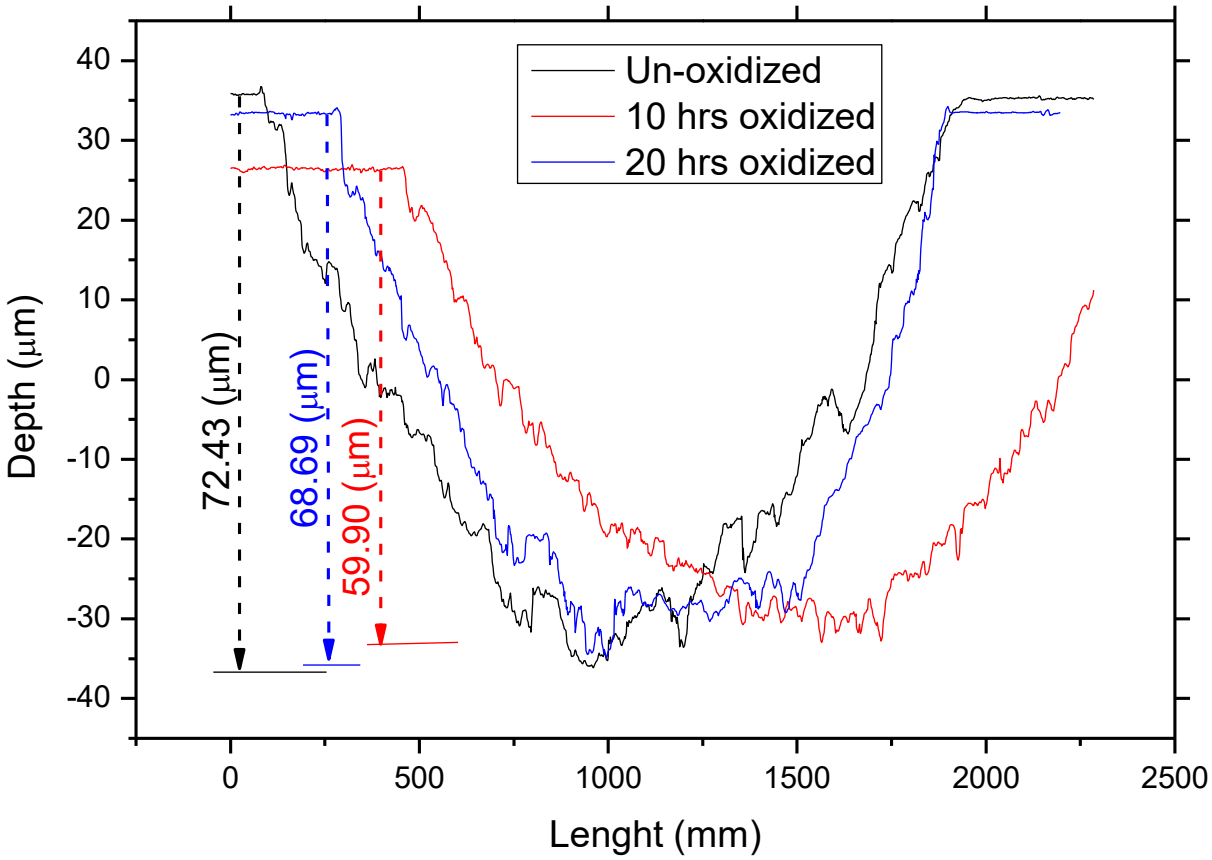
20 hrs oxidized Si_3N_4 + 3 wt. % MWCNTs



Coefficient of Friction (COF)

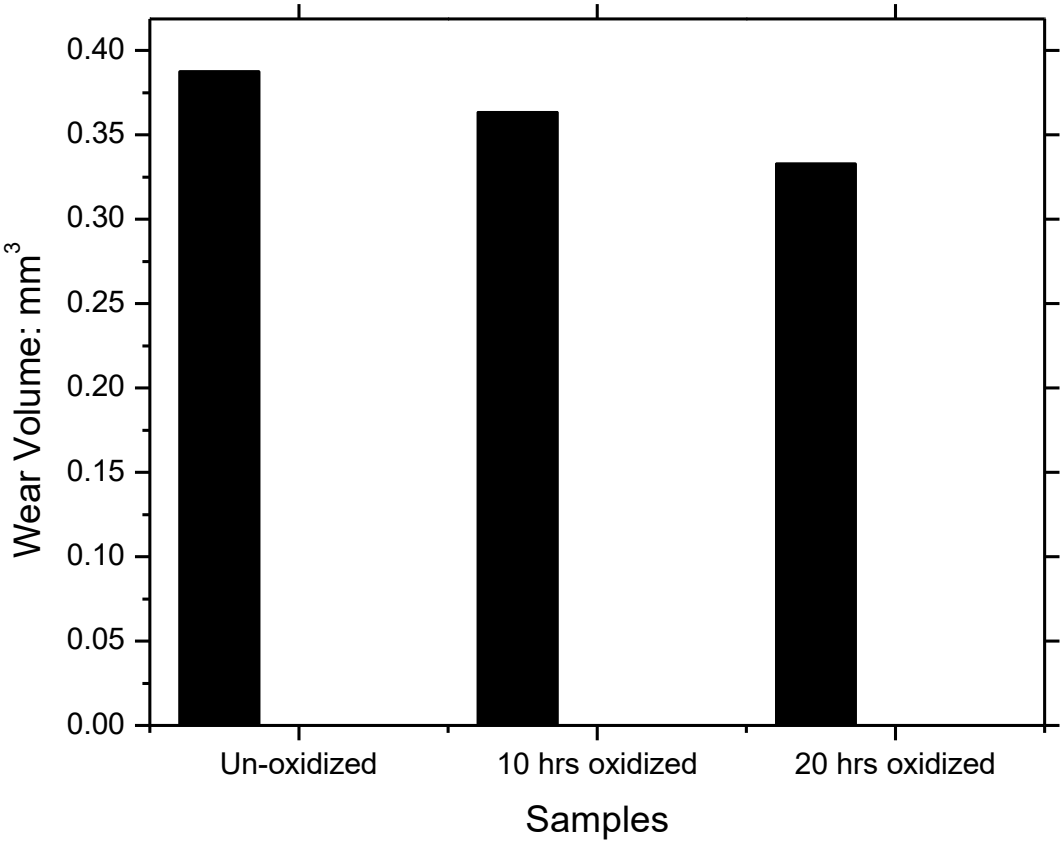


Depth of Track



Wear Volume

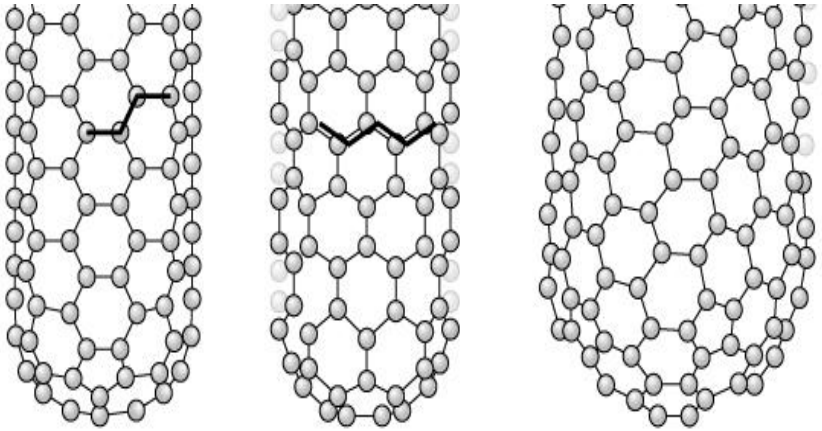
Decrease in wear volume is attributed to the relative density of the material. Higher relative density, lower wear volume



Silicon Nitride composites with the addition of CNTs – A review

Carbon Nanotubes

Material	Density (g/cm^3)	Tensile Strength (GPa)	Stiffness (GPa)
CNTs	1.3 – 2	10 – 60	1000
Wood	0.6	0.008	16
Steel	7.8	0.4	208
Carbon Fiber	1.7 – 2.2	1.7 – 3.3	200 – 960
Epoxy	1.25	0.005	3.5



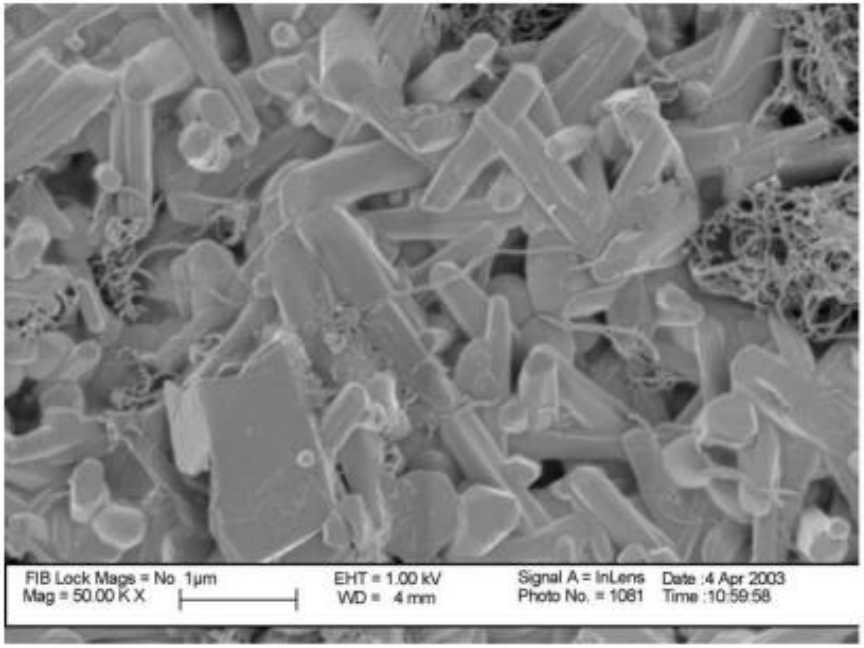
Si_3N_4

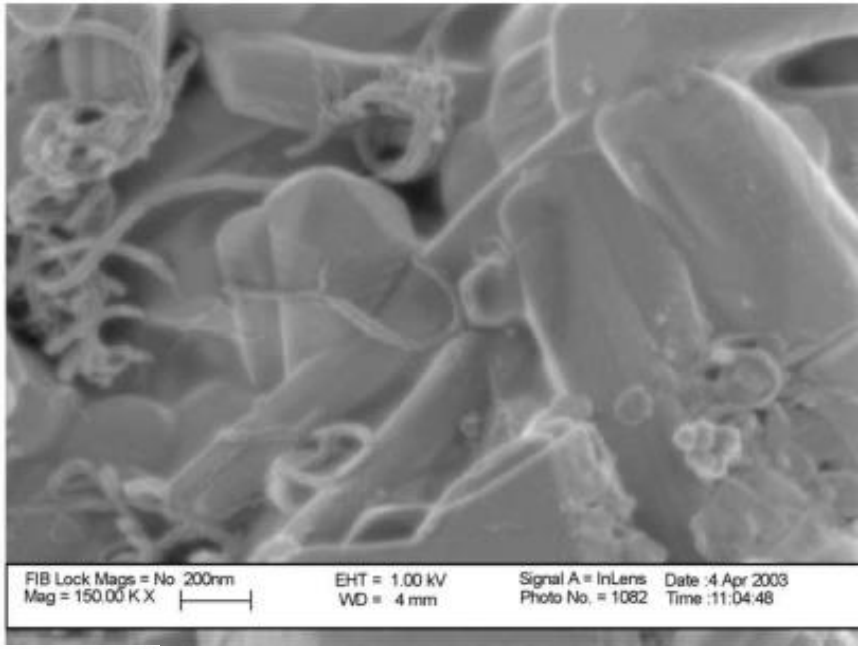
- Silicon nitride (Si_3N_4) is classified as an advanced structural ceramic with high melting point, hard and relatively chemical inert. It has three crystallographic structure on room temperature which are named as α, β and ϒ.
- cutting tools, bearings, sealings and gas turbine engines due to its exceptional flexural strength, high hardness, resistance to oxidation and thermal properties .
- brittleness, low flaw tolerance, limited number of slip systems and low reliability limit, low electrical conductivity its applications in several sectors.

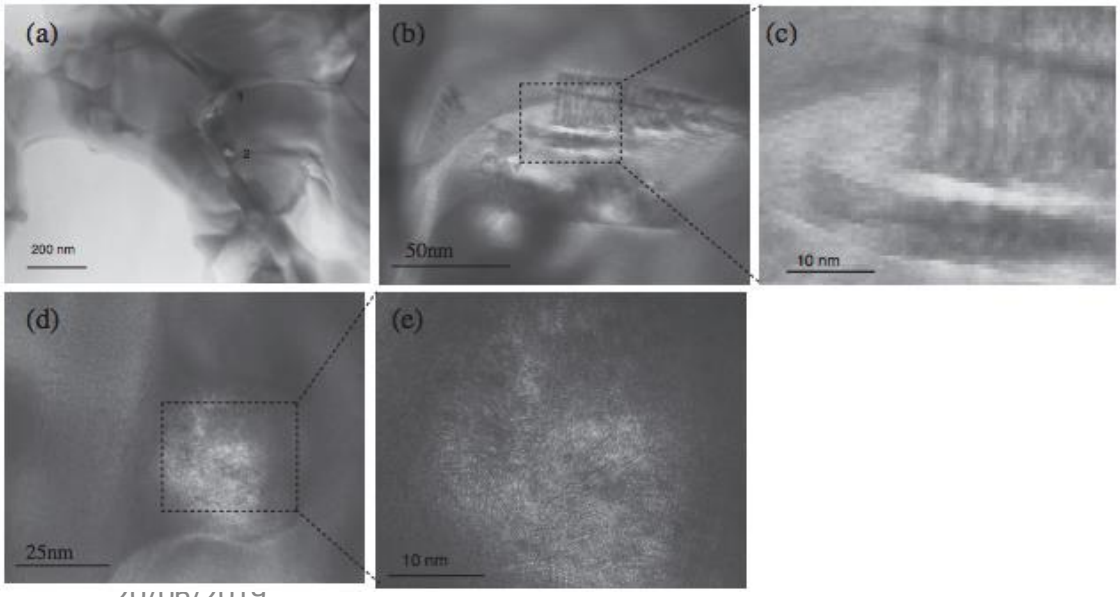
Processing of Si_3N_4+CNTs composites

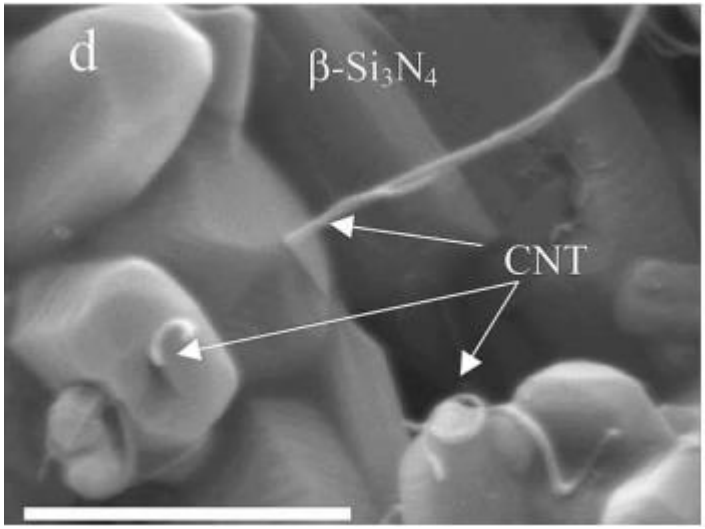
- Efficient milling process enhances the uniform dispersion of CNTs in the matrix and eventually uniform dispersion enhances the relative density of the sintered composites.
- Processing Techniques: Hot Isostatic Pressing (HIP), Hot pressing (HP), Gas pressure sintering (GPS), Spark Plasma Sintering (SPS).
- Sintering Additives: TiO_2, Y_2O_3, Al_2O_3, MgO, SiO_2, AlN, HfO_2 and ZrO_2

Microstructure Development

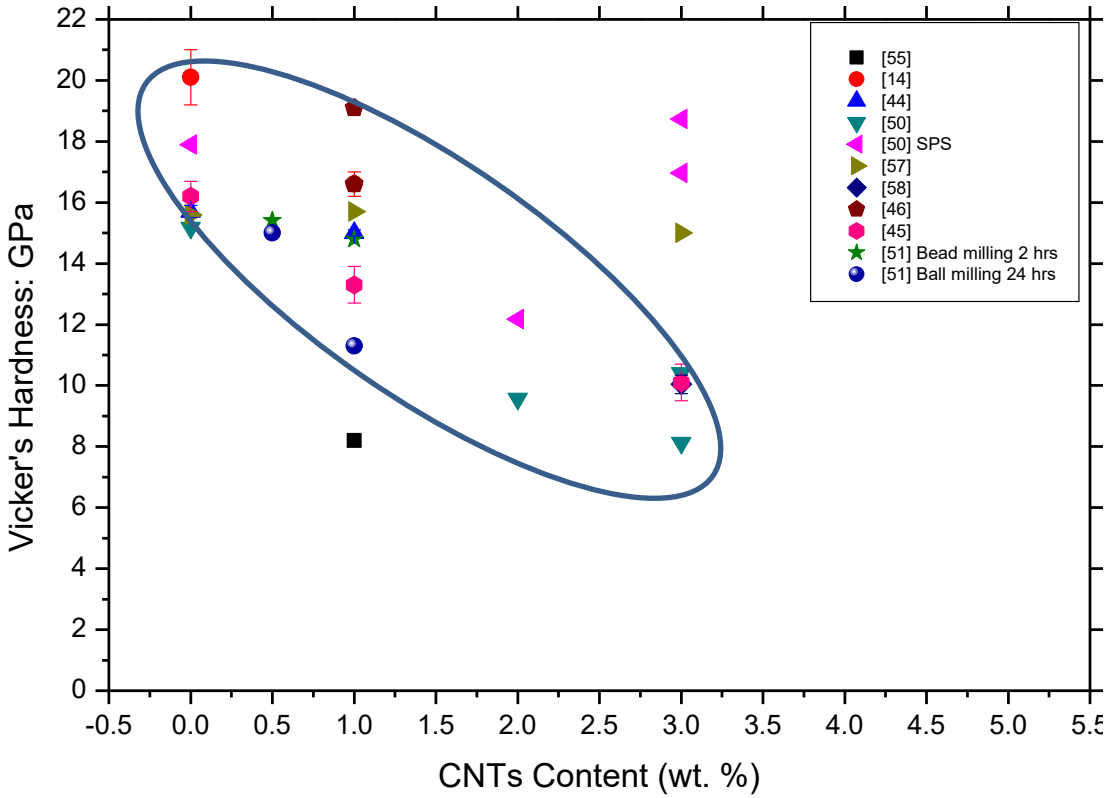




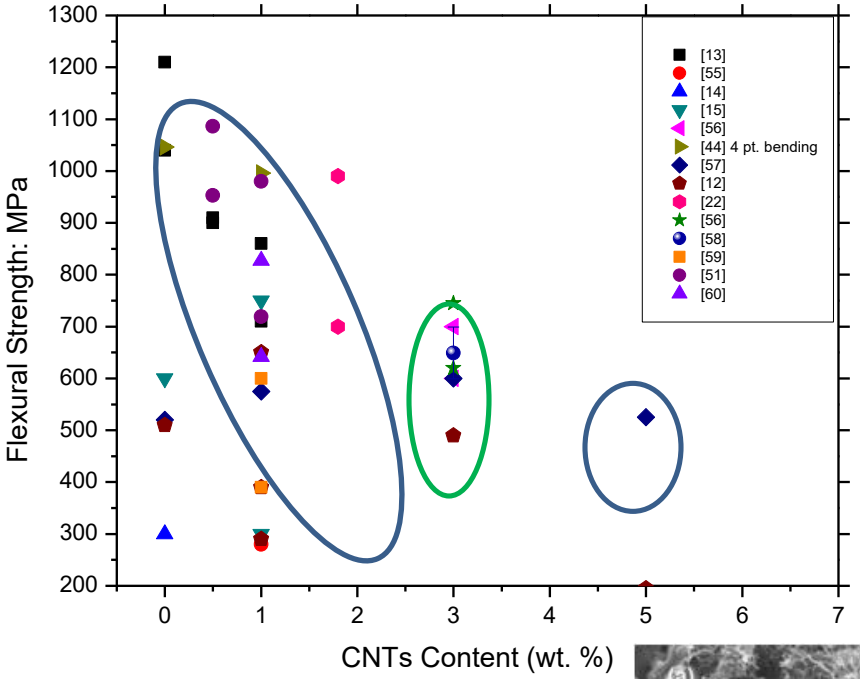


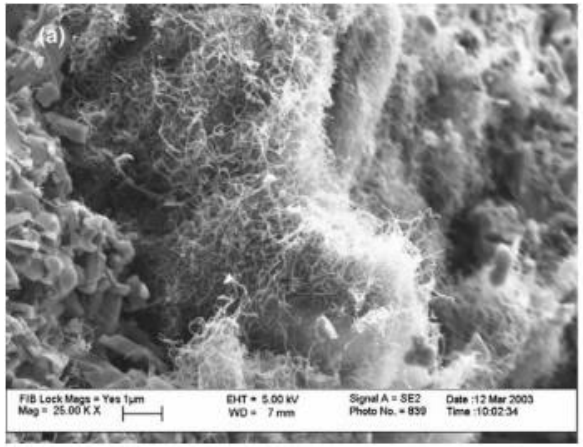


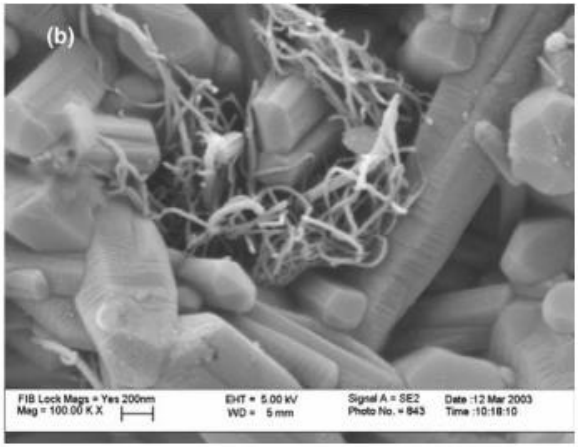
Influence on Hardness



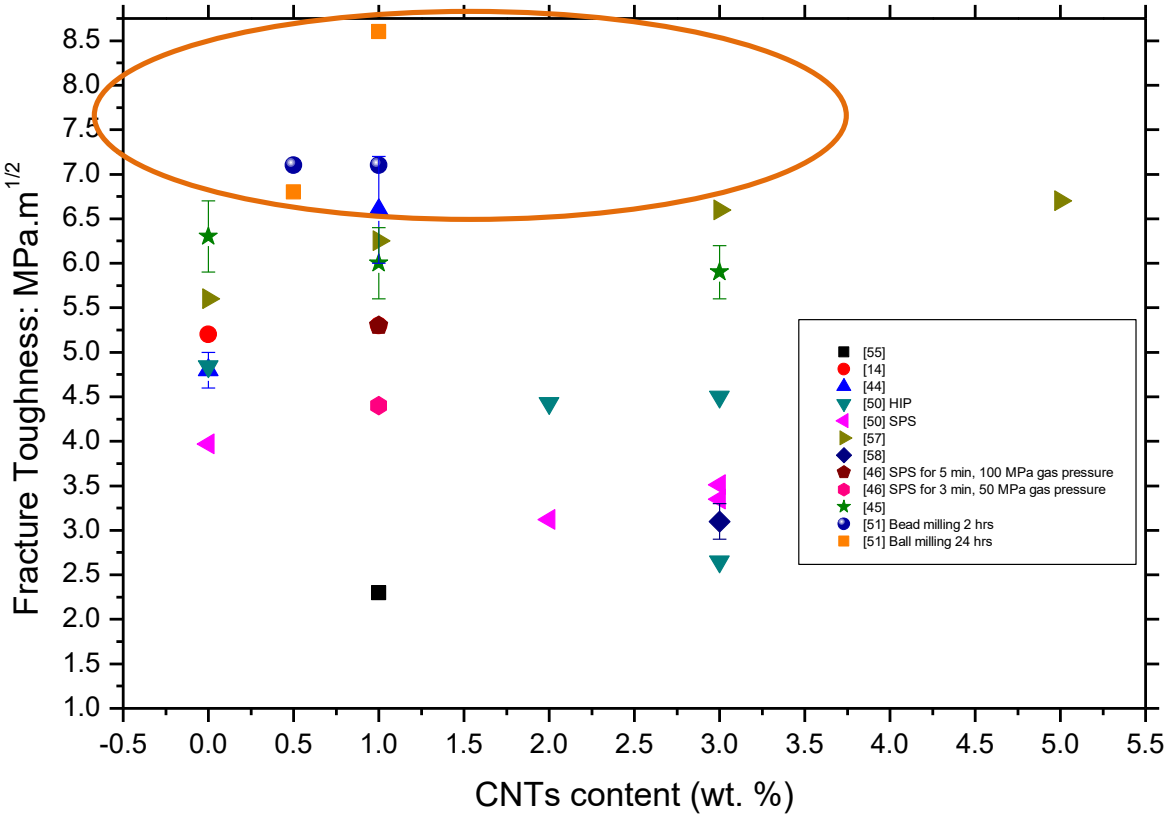
Influence on Strength





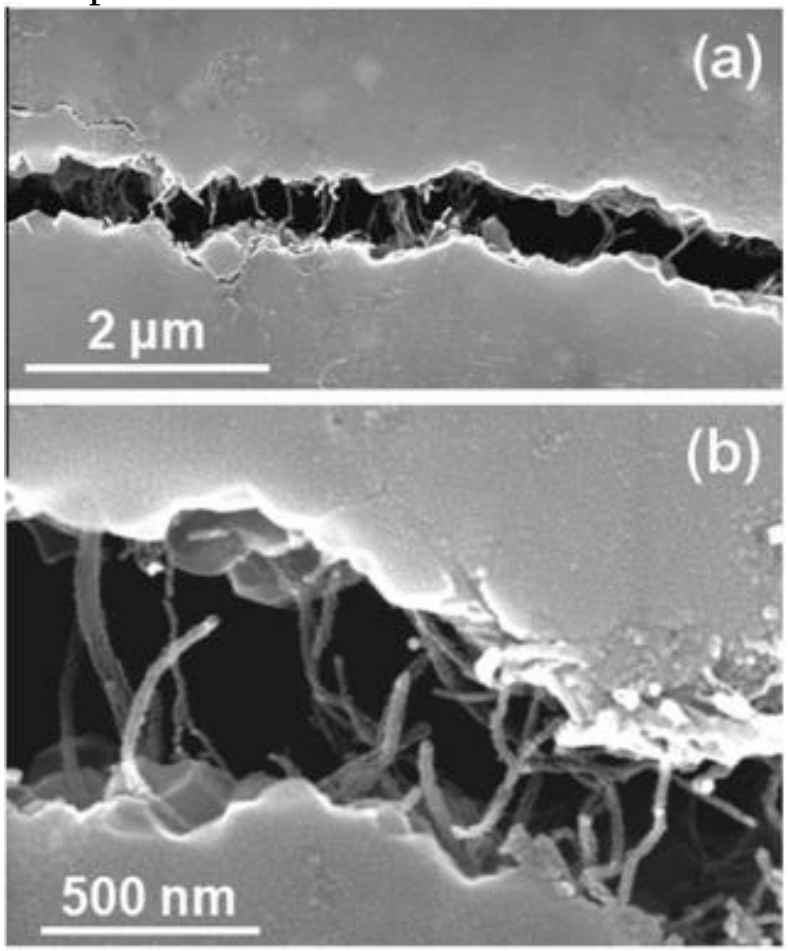


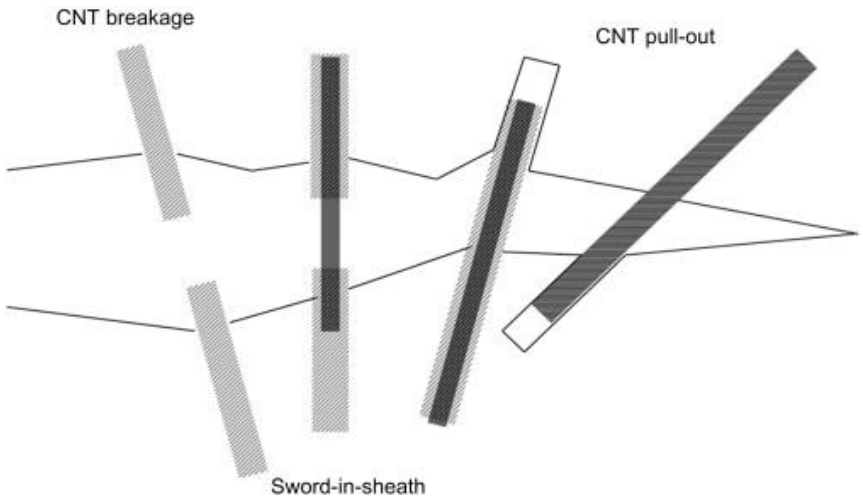
Influence on Fracture Toughness



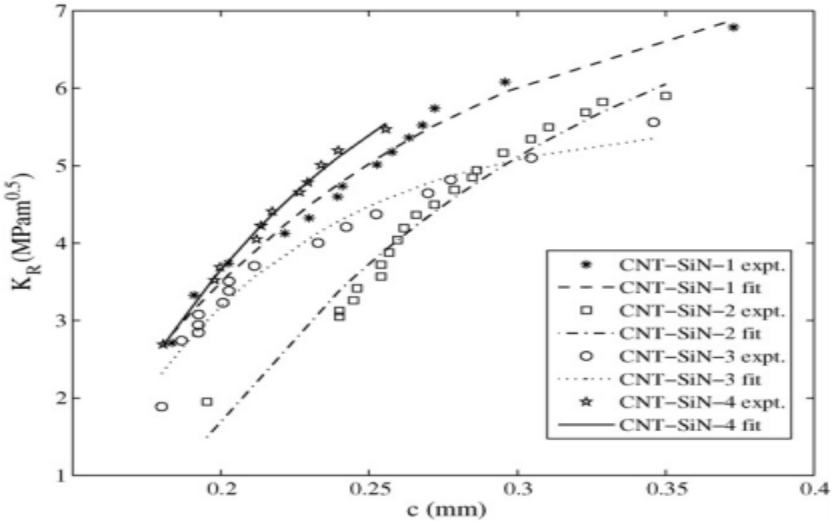
Toughening Mechanism by CNTs

Crack deflection at the CNT/ Si_3N_4 interface, crack-bridging by CNTs and CNTs pulling out on the fracture surface of silicon nitride composites



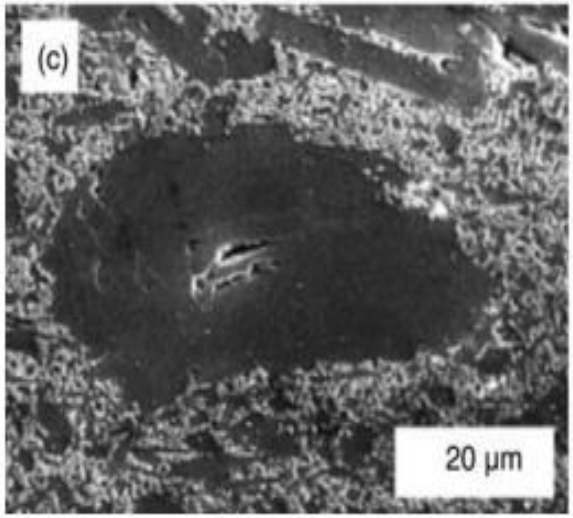


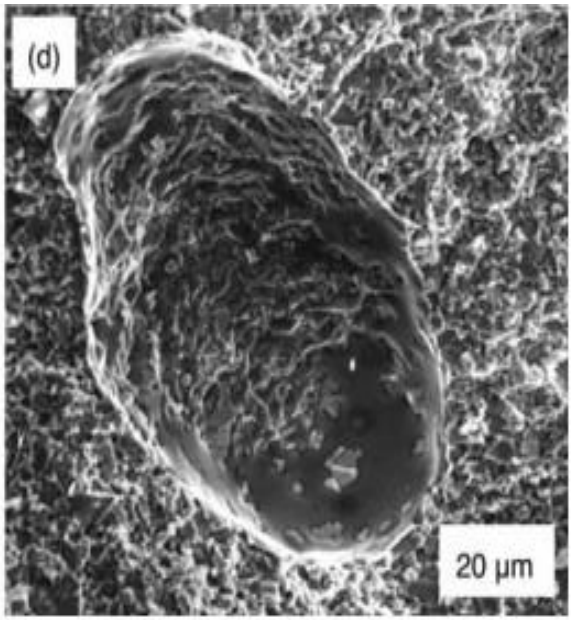
fracture toughness increases with the increasing size of the crack

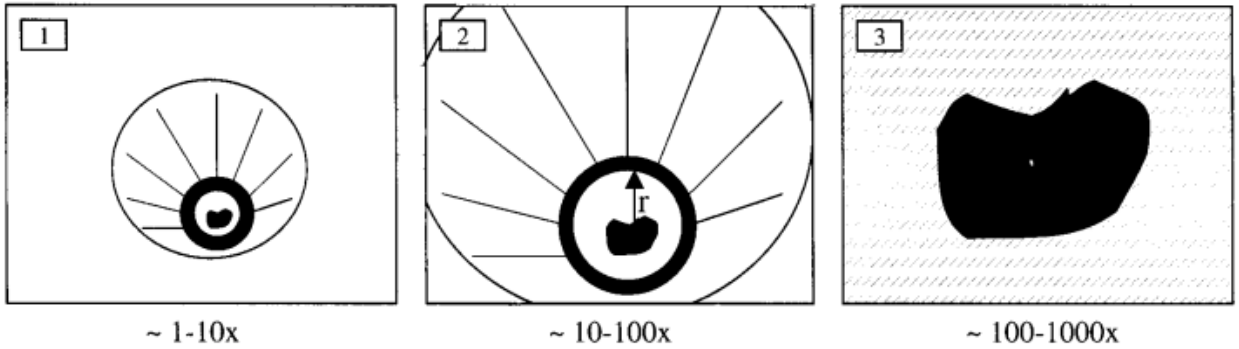


Origin of Fracture

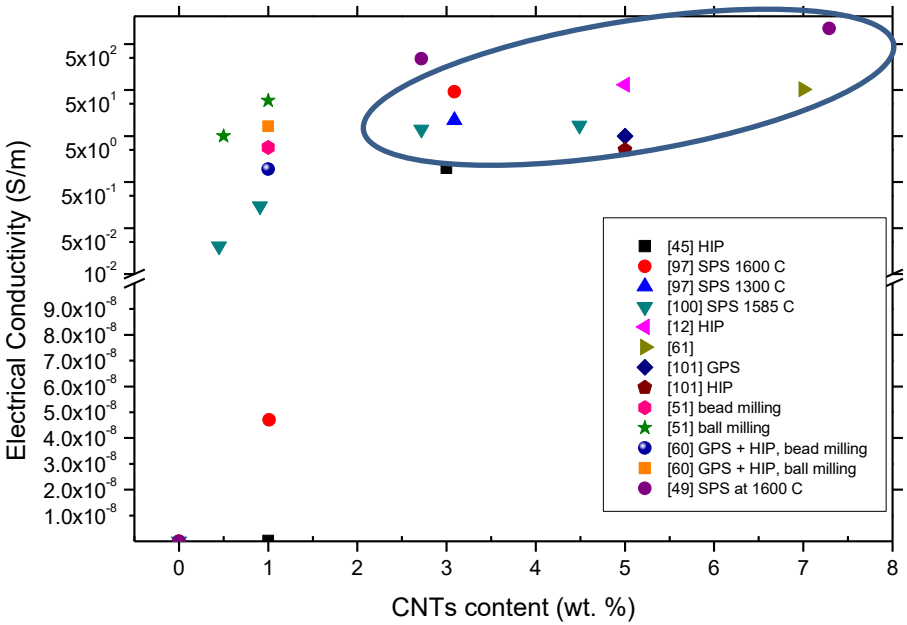
fracture origin is microstructure imperfections such as pores, inner residual cracks, non-densified part, clusters of reinforcement particles and impurities.



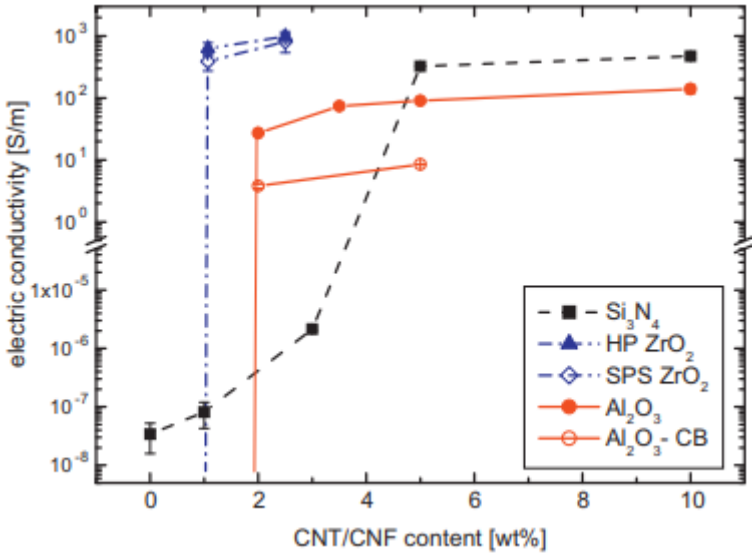




Electrical Properties



electrical resistivity of monolithic Si_3N_4 >10^{14} Ω.cm at room temp



Major challenges in integrating CNTs in Si_3N_4

- Uniform dispersion
- Damaging of CNTs
- Oxidation of CNTs
- Clustering of CNTs
- Densification inhibition
- CNTs induce porosities in the composite
- Poor interfacial boding between matrix grains and CNTs

Concluding Remarks

- CNTs induce the porosity which is main cause of degradation of mechanical propoerties
- CNTs can cuase of toughening mechanism by crack bridigng, crack deflection and pulling-out.
- Electrical properties are enhanced with the addtion of CNTs
- Tribological properties also enhanced with the addition of CNTs

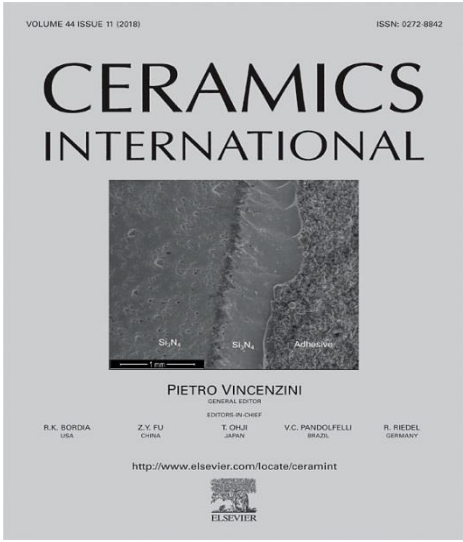
Concluding Remarks

- To obtain CNT-reinforced Si_3N_4 with better properties and tailored microstructure, these issues should be considered: achieving homogeneous dispersion of CNTs in the Si_3N_4 matrix, to avoid the CNTs agglomeration, entangling, clustering and damaging, interfacial bonding between CNT and Si_3N_4 grains and toughening mechanism (crack bridging, crack deflection and pull-out mechanisms).

Future Plans

- Oral presentation at 6th international conference ***"Fractography of Advanced Ceramics"*** in the Smolenice Castle Congres Center, Smolenice SAS on September 08 - 11, 2019.
- Tribological test under different loads
- Electrical Conductivity measurement of monolithic and CNTs reinforced Si_3N_4.
- Thermal Properties measurement of monolithic and CNTs reinforced Si_3N_4.
- Writing an article on Tribological properties of Si_3N_4+3 wt.% MWCNTs.
-

List of Publications 2017-2019



- ***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; Jan Dusza* "Processing and properties of S3N4 + MWCNTs composites from oxidized silicon nitride powder"**, July 2019 (to be submitted)** (Journal with IF).
- ***Awais Qadir****; Pinke Peter; Jan Dusza;* " CNTs reinforced silicon nitride composites - A review**, July 2019 (to be submitted)** (Journal with IF).

Participation in Conferences 2017-2019

- Junior EuroMat Conference 2018, Budapest
- Fine Ceramics Day 2018, Budapest
- Attended Hungarian Microscopic Conference 2017 in Siofok, Hungary - **Magyar Mikroszkópos Társaság**

- 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

References

- [14] Cs. Balázsi, Wéber, F., Kövér, Z., Shen, Z., Konya, Z., Kasztovszky, Z., ... & Arato "Application of carbon nanotubes to silicon nitride matrix reinforcements," *Curr. Appl. Phys.*, vol. 6, no. 2, pp. 124–130, Feb. 2006.
- [44] S. Pasupuleti, Peddetti, R., Santhanam, S., Jen, KP, Wing, ZN, Hecht, M., & Halloran, JP "Toughening behavior in a carbon nanotube reinforced silicon nitride composite," *Mater. Sci. Eng. A*, vol. 491, no. 1, pp. 224–229, Sep. 2008.
- [45] A. Kovalčíková, Cs. Balázsi, J. Dusza, and O. Tapasztó, "Mechanical properties and electrical conductivity in a carbon nanotube reinforced silicon nitride composite," *Ceram. Int.*, vol. 38, no. 1, pp. 527–533, Jan. 2012.
- [46] Cs. Balázsi, Shen, Z., Kónya, Z., Kasztovszky, Z., Weber, F., Vertesy, Z., ... & Arato, P. "Processing of carbon nanotube reinforced silicon nitride composites by spark plasma sintering," *Compos. Sci. Technol.*, vol. 65, no. 5, pp. 727–733, Apr. 2005.
- [47] E. L. Corral, Cesarano III, J., Shyam, A., Lara-Curzio, E., Bell, N., Stuecker, J., ... & Barrera, EV "Engineered Nanostructures for Multifunctional Single-Walled Carbon Nanotube Reinforced Silicon Nitride Nanocomposites," *J. Am. Ceram. Soc.*, vol. 91, no. 10, pp. 3129–3137, 2008.
- [48] J. Gonzalez-Julian, J. Schneider, P. Miranzo, M. I. Osendi, and M. Belmonte, "Enhanced Tribological Performance of Silicon Nitride-Based Materials by Adding Carbon Nanotubes," *J. Am. Ceram. Soc.*, vol. 94, no. 8, pp. 2542–2548, 2011.
- [49] M. Belmonte, S. M. Vega-Díaz, A. Morelos-Gómez, P. Miranzo, M. I. Osendi, and M. Terrones, "Nitrogen-doped-CNTs/Si3N4 nanocomposites with high electrical conductivity," *J. Eur. Ceram. Soc.*, vol. 34, no. 5, pp. 1097–1104, May 2014.
- [50] O. Tapasztó, Kun, P., Weber, F., Gergely, G., Balazsi, K., Pfeifer, J., ... & Balazsi, C. "Silicon nitride based nanocomposites produced by two different sintering methods," *Ceram. Int.*, vol. 37, no. 8, pp. 3457–3461, Dec. 2011.
- [51] M. Matsuoka, J. Tatami, T. Wakihara, K. Komeya, and T. Meguro, "Improvement of strength of carbon nanotube-dispersed Si3N4 ceramics by bead milling and adding lower-temperature sintering aids," *J. Asian Ceram. Soc.*, vol. 2, no. 3, pp. 199–203, Sep. 2014.
- [52] C. Balázsi, K. Sedláčková, and Z. Czigány, "Structural characterization of Si3N4–carbon nanotube interfaces by transmission electron microscopy," *Compos. Sci. Technol.*, vol. 68, no. 6, pp. 1596–1599, May 2008.
- [60] S. Yoshio *et al.*, "Dispersion of carbon nanotubes in ethanol by a bead milling process," *Carbon*, vol. 49, no. 13, pp. 4131–4137, Nov. 2011.
- [61] P. Ge, K. Sun, A. Li, and G. Pingji, "Improving the electrical and microwave absorbing properties of Si3N4 ceramics with carbon nanotube fibers," *Ceram. Int.*, vol. 44, no. 3, pp. 2727–2731, Feb. 2018.
- [97] E. L. Corral, H. Wang, J. Garay, Z. Munir, and E. V. Barrera, "Effect of single-walled carbon nanotubes on thermal and electrical properties of silicon nitride processed using spark plasma sintering," *J. Eur. Ceram. Soc.*, vol. 31, no. 3, pp. 391–400, Mar. 2011.
- [100] J. González-Julián, J., Iglesias, Y., Caballero, AC, Belmonte, M., Garzón, L., Ocal, C., ... & Osendi, "Multi-scale electrical response of silicon nitride/multi-walled carbon nanotubes composites," *Compos. Sci. Technol.*, vol. 71, no. 1, pp. 60–66, Jan. 2011.
- [101] B. Fényi, P. Arató, F. Wéber, N. Hegman, and C. Balázsi, "Electrical Examination of Silicon Nitride – Carbon Composites," *Materials Science Forum*, 2008. [Online]. Available: https://www.scientific.net/MSF.589.203. [Accessed: 15-May-2019].

Thank You for your attention!

Köszönöm a figyelmet!