Micro-mechanics Study of Nano TiO\textsubscript{2} High Friction Composite Material Using Pin on Disc Method

G K Kannan\textsuperscript{1*}, S.Stephen Bernard\textsuperscript{2}, L.Ranjith\textsuperscript{3}, M.V.Jagadeep\textsuperscript{1}, S.Jawahar Ganesh\textsuperscript{3}, and S.Jawahar Santhosh\textsuperscript{1}

\textsuperscript{1}Department of Mechanical Engineering, Chennai Institute of Technology, Chennai - 600069, Tamil Nadu, India.
\textsuperscript{2}Department of Mechanical Engineering, Rajalakshmi Institute of Technology, Chennai - 600 124, Tamilnadu, India.
\textsuperscript{*}Corresponding author mail ID: gkkannan.85@gmail.com

Abstract: This paper focuses with the analysis of friction and abrasion characteristics of Nano TiO\textsubscript{2} composite using pin on disc method. Nano TiO\textsubscript{2} with 10 ingredients was fabricated by employing compression moulding technique and also the composite tribology strength was weighed. The six composite specimens were prepared by varying Nano TiO\textsubscript{2} (2, 4, 6, 8 and 10\%) with other ingredients and the results are plotted. It indicates that the abrasion rate was decreased by adding Nano TiO\textsubscript{2} upto 8\%. Hardness and porosity are also found to increase but heat swell and specific gravity decreases. 6 wt\% Nano TiO\textsubscript{2} has the property to improve the friction stability during high speed and load. Abrasion tested composites was examined by scanning electron microscope indicating that abrasive, cavity and oxidation were the predominant abrasion mechanism. [Copyright information to be updated in production process]

Keywords: Nano TiO\textsubscript{2}; Friction; Abrasion; SEM; Pin on Disc; EDX

1. Introduction
High friction composite materials are used in many applications like brake pad, Clutch plate etc. Generally high friction materials are not monolithic. It is fabricated by more than 10 ingredients to attain high friction stability and good abrasion resistance [1]. A friction material necessitates a mixture of lubrication and friction additives, reinforcement, binder and filler. Out of these four necessities, friction additives plays the major role of abrasion resistance at high load and speed and they show exemplary friction quality when contact is made with two surfaces [2, 3]. Nano TiO\textsubscript{2} has given more attention in many areas because of their structure, resistance to bonding, low cost, high surface, non-toxic, elementary composition and high absorption factors [4]. Structural formation and glassy interface are two factors that influence TiO\textsubscript{2} during composite manufacture. Nano TiO\textsubscript{2} are mainly used in cosmetic products, sensor, medicine wrapper, laptop case, paper, catalysis, solar cell transducer, glass ceramics etc [5].

A quantity of Nano TiO\textsubscript{2} present in friction composite significantly depends on the method of manufacturing and their chemical compatibility with the constituent matrix material [6]. Nano TiO\textsubscript{2} acts as a driving force for the utilization of metal matrix composites by its performance, low cost and environmental benefits. Nano TiO\textsubscript{2} exists in three different crystalline phases, they are one stable phase and two metastable polymorph phase. Metastable phase are rutile (tetragonal) and metastable phase are anatase (tetragonal) and brookite (orthorhombic) [7, 8]. The three phases have entirely different physical and chemical properties e.g., photochemical reactivity, dielectric constant and refractive index.

The examination on abrasion, contact and method of cooling amid test-piece connect gives significance due to their work of constrained mating on a surface. In the middle of connection of two diverse materials, poor quality specimen will encounter a material mismatch from a connected plane and plane movement of resistance. Tribological tests are utilized to degree the factor of drag and abrasion of the composites amid their introductory advancement since it is as well expensive to test the composites for specific application testing pack [9]. Utilizing pin on disc tribology and abrasion
readings compare to idleness dynamometer test but the estimate of different surrounding state may not be attained. This work is on mechanical resistance of Nano TiO$_2$ is weighed using Win-DUCOM friction tester and the SEM is analysed for 1000 rpm for all samples.

2. Materials and Methodology

For production of composite material, all the ingredients were purchased. Six different proportion of Nano TiO$_2$ composites are fabricated to analyse the mechanical and tribological property of the specimen are shown in Table 1. Nano TiO$_2$ and vermiculate are varied for producing composite and the remaining ingredients kept constant. The weight fraction of Nano TiO$_2$ is determined by the proportion of the weight of Nano TiO$_2$ to the total weight of the mixture. The various proportions are tabulated in Table 1. The DUCOM instrument was used to weigh the friction characteristics and abrasion volume loss. The counter material is C.I, and the tested specimen is illustrated in Fig. 1. A data gathering system automatically enters the readings. The counter material is round Cast Iron disc and the tested specimen size is 8mm length and 32mm diameter. ASTM G-99 standard procedure is followed for testing the specimen. By varying speed (250, 500, 750, 1000 rpm) for 49.5 N up to a sliding distance of 3000 m, the tests were carried out.

For production of composite material, all the ingredients were purchased. Six different proportion of Nano TiO$_2$ composites are fabricated to analyse the mechanical and tribological property of the specimen are shown in Table 1. Nano TiO$_2$ and vermiculate are varied for producing composite and the remaining ingredients kept constant. The weight fraction of Nano TiO$_2$ is weighed by the proportion of the mass of Nano TiO$_2$ to the total mass of the mixture. The various proportions are tabulated in Table 1. The DUCOM instrument was used to weigh the friction characteristics and abrasion volume loss. The counter material is C.I, and the tested specimen is illustrated in Fig. 1. A data gathering system automatically enters the readings. The counter material is round Cast Iron disc and the tested specimen size is 8mm length and 32mm diameter. ASTM G-99 standard procedure is followed for testing the specimen. By varying speed (250, 500, 750, 1000 rpm) for 49.5 N up to a sliding distance of 3000 m, the tests were carried out.

| Table 1 Preparation of friction material by varying cardanol content |
|-------------------|-----|-----|-----|-----|-----|-----|
| Contents (Wt %)   | S1  | S2  | S3  | S4  | S5  | S6  |
| Nano TiO$_2$      | 0   | 2   | 4   | 6   | 8   | 10  |
| Vermiculate       | 10  | 8   | 6   | 4   | 2   | 0   |
| Phenolic Resin    | 20  | 20  | 20  | 20  | 20  | 20  |
| CaCO$_3$          | 15  | 15  | 15  | 15  | 15  | 15  |
| Antimony Trisulfide | 12 | 12  | 12  | 12  | 12  | 12  |
| Graphite          | 6   | 6   | 6   | 6   | 6   | 6   |
| Cashew dust       | 14  | 14  | 14  | 14  | 14  | 14  |
| Steel Fiber       | 10  | 10  | 10  | 10  | 10  | 10  |
| Silicon           | 3   | 3   | 3   | 3   | 3   | 3   |
| Alumina           | 10  | 10  | 10  | 10  | 10  | 10  |
Figure 1. (a) Tribology Experimental Set up with data acquisition

(b) Tested Samples with C.I Disc

Factor of drag ($\mu$) of the sample was evaluated by the equation

$$\mu = \frac{F_f}{F_n}$$  \hspace{1cm} (1)

Where, $F_n$ is the applied load and $F_f$ is the average Drag force.

Friction Stability ($F.S$) evaluated using the below equation

$$F.S = \frac{\mu_{avg}}{\mu_{max}} \times 100$$  \hspace{1cm} (2)

Where, $\mu_{max}$ is the maximum COF and $\mu_{avg}$ is the average COF.

3. Results and Discussion

Fig. 2. indicates the mechanical property of five different proportion Nano TiO$_2$ composites compared with vermiculate composite. It clearly indicates that the hardness value of 63 is weighed for vermiculate composite (S 1), which gradually increases (68, 72, 75, 78) with the introduction of Nano TiO$_2$ particles. This increment is due to the hardness transference and increased density from vermiculate to the Nano TiO$_2$ reinforced particles that provide to increase the hardness [10]. Loss on Ignition increases by increasing the Nano TiO$_2$. This may be due to the ceramic nature of the Nano TiO$_2$ which resists the loss during ignition. Heat swell decreases by increasing the Nano TiO$_2$ due to resistance of internal plasticizing effect of the ingredients [11]. Density reduces with the rise in the content of Nano TiO$_2$ particles. This is on the grounds that Nano TiO$_2$ owns high density particles compared to vermiculate. It is noted from the above statistics that the specific gravity reduces with the rise in the content of Nano TiO$_2$ particles. The rejection of particles during compression moulding can be credited to this phenomenon. This is on the grounds that Nano TiO$_2$ owns low specific gravity.
compared to vermiculate to settle down in compression moulding [12]. The specific gravity was found to diminish from 2.36 to 2.2 with Nano TiO2 expanding from 2% to 10% due to a lessening of void division.

![Figure 2. Properties of Nano TiO2 composite friction specimen](image)

**Figure 2.** Properties of Nano TiO2 composite friction specimen

![Figure 3. Abrasion Rate of Nano TiO2 composite](image)

**Figure 3.** Abrasion Rate of Nano TiO2 composite for (a) 250 rpm (b) 500 rpm (c) 750 rpm and (d) 1000 rpm.

It is obvious from the Fig. 3, that the abrasion rate drastically decreasing with the increment within the sliding distance at each stack connected. By increasing speed to 500 rpm leads to the diminish
within the abrasion rate by 12% compared to 250 rpm, by which it is clear that the higher Nano TiO\textsubscript{2} ingredients increase the hardness of the material [13].

The matrix of the composite surface has been guarded from the counter surface (Nano TiO\textsubscript{2}) by the difficult asperities display within the composites. Hence the beginning run and the relentless state condition of the sliding prepare have appeared critical change in abrasion rate [14]. Due to the increase in the interfacial area by the Nano TiO\textsubscript{2} particles, the resistance to dislocations and the load bearing capacity of the material has been significantly increased. The difficult grating ensures the lattice, which leads to the reduced abrasion rate within the beginning run. This gathers that the including of Nano particles with binder is not affected by increase the speed. When the counter surface speed is expanded from 500 to 1000 rpm, the abrasion rate is diminished to 16.6%. This may be due to intermetallic stage which is shaped by the solid interfacial bond between the matrix and the Nano TiO\textsubscript{2} shows higher abrasion resistance [15]. Fig. 3d.indicates the specimen property increases the abrasion resistance due to the strength provided by the uniform distribution of particles. And also the abrasion loss is controlled by the formation of oxide layer between the two solid surfaces.

![Figure 4](image_url)

**Figure 4.** Average Factor of drag for Nano TiO\textsubscript{2} composite for varying speed

Average Factor of drag for Nano TiO\textsubscript{2} composite for varying speed is shown in Fig. 4. Average COF of Nano TiO\textsubscript{2} composite has been found higher than vermiculate composite. Figure clearly indicates that by adding Nano TiO\textsubscript{2} average COF value is increase for all the speed. However, as 750 rpm , sudden drop of COF is observed indicating that high composition of Nano TiO\textsubscript{2} at high speed leads to damage the counter Cast Iron disc. This leads to abrasion loss in a high friction composite specimen.

By increasing the speed from low (250 rpm) to high (1000 rpm), the factor of drag is increased for all specimens. It happens due to genuine region of contact being expanded unreasonably and excessively since, ceramic materials are non viscous. Diminish in COF may moreover be due to debasement of ceramic at the harsh contacts driving to era of carbon dioxide [16].

The leading factor of drag found for S2, S3 and S5 composite may be due to tall consideration of Nano TiO2 filaments with restricted amount of vermiculate. This too might be the reason to not formation of cold welding and crack of asperities of the composite surfaces. Consequently, there are no withdrawn asperities caught between the sliding planes which might have caused an increment within the factor of contact to a optimum level. With increment in speed S 5 and S 6 appeared over the top increment COF affirming its tall affectability of COF for speed. For rest of grinding fabric there was no other obvious contrast within the grinding factor.

Fig.5a. shows white patches, i.e. oxide layer formation on the layer. Also micro cracks were observed at the edge of the white patch which indicates the surface elimination process with the aid of using the propagation of cracks below improved stress. It is specific that white patches are fashioned because of ploughing within side the sample’s surface. The white patches formed due to ploughing leads to weak points around it. This weak point obviously leads to cracking of the surface. Fig.5b. shows that the surface has cavities in it and the asperities have adjusted parallel to the course of sliding. The cavities were shaped due to the eliminated particles amid sliding. Plastic deformation has been found on the edges of the fine grooves within the abrasion surfaces. Low density materials widely employed in automobile and aerospace applications [17-28].
Figure-5c. and 5d. shows the cavity and adhesive abrasion appears on the worn surface alongside the crack lines while at high speeds the cavitation tends to reduce but deep cracks and grooves are clearly visible. Plastic strains developed in the composite makes in a contact with the steel counter face leads to subsurface crack propagation.

The three body abrasion ranges are found at the boundaries of worn surface. This may be due to strain solidifying of Nano TiO$_2$ amid sliding with a speed additionally due to embrittlement of difficult particles amid sliding. It is found that amid sliding cast press counter face steadily crushes the difficult particulate and lean oxidized layer is shaped on the composite surface which carries on as a lubricant that leads to alter the basic appearance of the composite.

Blow hole generally formed on the surface of the specimen during high impact of the counter disc. Abrasion surface creates the hard particles to leave which form the blow hole. This generally happens at higher rpm of rotating disc. Fig. 5f. Clearly indicates the blow hole which is formed near to the third body abrasion. This may be due to the fault on the specimen preparation. High interest should be given during fabrication. High pressure and temperature maintaining for the production of high friction material plays the major role for formation of blow hole.

![Figure 5c](image1)
![Figure 5d](image2)

Figure. 5. SEM image of abrasion samples (a) S 1 @ 1000 rpm (b) S 2 @ 1000 rpm (c) S 3 @ 1000 rpm (d) S 4 @ 1000 rpm (e) S 5 @ 1000 rpm (f) S 6 @ 1000 rpm
4. Conclusions
The following conclusion were found from the experiment and mentioned below

The characterizations of Nano TiO$_2$ particle dispersed in a high friction composite were also studied using SEM analysis. At the edge of the sliding, micro cracks have been observed, illustrating the mechanism of material removal by propagating cracks under increased stress.

All the six specimen showed the CoF value between 0.34 to 0.43, which lies in the standard range (0.3 to 0.45) of high friction composite material. The friction factor increased with speed for all the composites.

References
[1]. Jayashree Bijwe, Mukesh Kumar, “Optimization of steel wool contents in non-asbestos organic (NAO) friction composites for best combination of thermal conductivity and tribo-performance”, Abrasion. 2007; 263: 1243-1248. doi.org/10.1016/j.abrasion.2007.01.125
[2]. Stephen Bernard S, Javeed Ahmed M, Dasaparaksh J, Saroj Nitin MR, Vivek S, Kannan GK, “Friction and Abrasion Properties of Bio-Based Abrasive in a High-Friction Composite Material” Advances in Manufacturing Technology. Lecture Notes in Mechanical Engineering. 2019; 577-585. https://doi.org/10.1007/978-981-13-6374-0_63
[3]. G.Suresh, “A study of sliding abrasion behavior of carbon fiber reinforced IPN composites”, Materials today : Proceedings, https://doi.org/10.1016/j.matpr.2020.05.124
[4]. G.Sai Krishnan, “Role of metal composite alloys in non-asbestos brake friction materials-A solution for copper replacement”, Materials today : Proceedings, https://doi.org/10.1016/j.matpr.2020.02.943
[5]. R. Kalidoss, S. Umapathy, Y. Sivalingam, “An investigation of GO-SnO$_2$-TiO$_2$ ternary nanocomposite for the detection of acetone in diabetes mellitus patient’s breath” Applied Surface Science, 2018; 449; 677-684.
[6]. Stephen Bernard S, Jayakumari LS. Pressure and Temperature Sensitivity Analysis of Palm fiber as a biobased reinforcement material in brake pad. Journal of the Brazilian Society of Mechanical Science and Engineering. 2018; 40; 152. https://doi.org/10.1007/s40430-018-1081-0
[7]. S.Rajamahendran, “An analysis on mechanical and sliding abrasion behavior of E-Glass fiber reinforced IPN composites”, Materials today : Proceedings, https://doi.org/10.1016/j.matpr.2020.07.072
[8]. G.Saikrishnan, “Investigation on the mechanical and morphological properties of red banana/ramiie fiber vinyl ester composites, IOP Conference Series: Materials Science and Engineering, https://doi.org/10.1088/1757-899X/961/1/012015
[9]. Stephen Bernard S, Jayakumari LS. Effect of the Properties of Natural Resin Binder in a High Friction Composite Material. Polimeros- Ciencia Tecnologia. 2014. 24; 2: 149-152. dx.doi.org/10.4322/polimeros.2014.038
[10]. Sai Krishnan G, Jayakumari LS, “Investigation on the physical, mechanical and tribological properties of areca sheath fibers for brake pad applications” Materials Research Express, 2019; 6: 085-109. https://doi.org/10.1088/2053-1591/ab2615
[11]. Minakshi Sultania, Rai, JSP, Deepakshrivastava, “Process modeling, optimization and analysis of esterification reaction of cashew nut shell liquid (cnsl)-derived epoxy resin using response surface methodology”.Journal of hazardous materials. 2011; 185: 1198–1204. https://doi.org/10.1016/j.jhazmat.2010.10.031
[12]. Stephen Bernard S, Jayakumari LS. Effect of Rockwool and Steel fiber on the Friction performance of brake lining materials. Materia rio de janeiro, 2016; 21: 656-665. https://doi.org/10.1590/S1517-707620160003.0063
[13]. Vimalanathan, P., Suresh, G., Rajesh, M, et al. “A Study on Mechanical and Morphological Analysis of Banana/Sisal Fiber Reinforced IPN Composites”. Fibers Polymer. 2021. https://doi.org/10.1007/s12221-021-0917-x
[14]. P. Ramesh, “Investigation on the physical/mechanical properties of NAO brake friction composites by using kenaf fiber”, IOP Conference Series: Materials Science and Engineering, https://doi.org/10.1088/1757-899X/961/1/012016

[15]. Vivek S, Jayakumari L.S, Stephen Bernard S, Suresh G, Javeed Ahmed Md, Arul Murugan S. “Tribological and mechanical properties of biobased reinforcement in a friction composite material”. Materia rio de janeiro, 2020. 25; 3: http://dx.doi.org/10.1590/s1517-707620200003.1085.

[16]. Yun Rongping, Filip Peter, Lu Yafei, “Performance and evaluation of eco-friendly brake friction materials”. Tribology International. 2010; 43: 2010-2019. doi.org/10.1016/j.triboint.2010.05.001.

[17]. Jayashree Bijwe, Mukesh Kumar, “Optimization of steel wool contents in non-asbestos organic (NAO) friction composites for best combination of thermal conductivity and tribo-performance”. Abrasion. 2007; 263: 1243-1248. doi.org/10.1016/j.abrasion.2007.01.125.

[18]. A. Rohit Sai Krishna, B. Vamshi Krishna, T. Sashank, D. Harshith, Ram Subbiah, 2020, Influence and assessment of mechanical properties on treated P91 steel with normalizing processes, Materials Today: Proceedings. 27, 2, 1555-1558.

[19]. K. Manjith Srinivas, S. Bharath, P. N. V. Krishna Chaitanya, M. Pramod, Ram Subbiah, 2020, Improving Tribological properties of P91 steels through carburizing process, Materials Today: Proceedings. 27, 2, 1575-1578.

[20]. Mohanavel, V., Ravichandran, M.: Experimental investigation on mechanical properties of AA7075-AlN composites. Mater Test. 61 (6), 554-558 (2019).

[21]. Mohanavel, V., Rajan, K., Ravichandran, M.: Synthesis, characterization and properties of stir cast AA6351-aluminium nitride (AlN) composites, Journal of Materials Research, 31 (2), 3824-3831 (2016).

[22]. V.Mohanavel, S.Suresh Kumar, J.Vairamuthu, P.Ganeshan, B.NagarajaGanesh, 2020, Influence of stacking sequence and fiber content on the mechanical properties of natural and synthetic fibers reinforced penta-layered hybrid composites, Journal of Natural Fibers, DOI : 10.1080/15440478.2021.1875368

[23]. Vinayagam Mohanavel, Thandavamoorthy Raja, Anshul Yadav, Manickam Ravichandran, Jerzy Winczek, Evaluation of Mechanical and Thermal Properties of Jute and Ramie Reinforced Epoxy-based Hybrid Composites, Journal of Natural Fibers, DOI : 10.1080/15440478.2021.1958432

[24]. B. Chaitanya Kumar, P. Sri Charan, Kanishkar Jayakumar, D. Alankrutha, G. Sindhu, Ram Subbiah, 2020, Assessment Of Wear Properties On Low Temperature Molten Salt Bath Nitriding On Austenitic Stainless Steel, Materials Today: Proceedings. 27, 2, 1541-1544.

[25]. Vinayagam Mohanavel, K.S.Ashraff Ali, S.Prasath, S.Tathish, 2020, Microstructural and tribological characteristics of AA6351/Si3N4 composites manufactured by stir casting, Journal of Materials Research and Technology, 9 (6), 14662-14672.

[26]. T. Lakshmi Deepak, G. Ananda Mithra, K. Lokesh, B. Sai Chandra, Ram Subbiah, 2020, Stability Of Expanded Austenite By Gas Nitriding Process On Austenitic Stainless Steel Material Under Low Temperature Conditions, Materials Today: Proceedings. 27, 2, 2020, 1681-1684.

[27]. K. Raja, B. Prabu, P. Ganeshan, V. S. Chandra Sekar & B. NagarajaGanesh, 2020, Characterization Studies of Natural Cellulosic Fibers Extracted from Shwetark Stem, Journal of Natural Fibers, DOI: 10.1080/15440478.2019.1710650

[28]. Ram Subbiah, Md. Rahel, A Sravika, R.Ambika, A.Srujana, E.Navya, 2019, Investigation on Microstructure and Mechanical Properties of P91 Alloy Steel Treated With Normalizing Process - A Review, Materials Today: Proceedings, 18, 7, 2265-2269.