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Effect of potassium titanate fibres and metal sulphides in the tribological behaviour of brake friction composites

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Abstract

In a brake system, brake friction composites are essential for converting kinetic energy into frictional heat. An optimal brake friction composite should ensure a stable coefficient of friction under extreme operational conditions. This study examines the tribological performance of brake friction composites incorporating potassium titanate fibres and metal sulphide lubricants in standard brake pad formulation. Brake friction composites were engineered by varying the potassium titanate fibre content from 10 to 30 %, while the lubricant mixture (comprising graphite, MoS_2, Bi_2S_3 and Sb_2O_3) was proportionally adjusted from 27 to 7 %, with the filler and binder concentrations held constant throughout the formulation. The phenolic resin-based composites were characterised for their physical, chemical and mechanical properties in accordance with the IS 2742 (Part 4) standard. The tribological performance of the fabricated composites was evaluated using a rotating drum setup, following the SAE J661 procedure. The worn surfaces of the samples were analysed to identify wear mechanisms utilising scanning electron microscopy (SEM) coupled with energy-dispersive X-ray spectroscopy (EDS). The results demonstrated that the optimal combination of potassium titanate fibres and metal sulphide lubricants significantly influences wear resistance and friction stability, with varying degrees of primary and secondary plateau formation observed. This comprehensive characterisation provides valuable insights into the material's behaviour under operational conditions, guiding the development of advanced brake friction composites.

1. Introduction

In the realm of automotive engineering, the transformation of kinetic energy into frictional heat during the braking process stands as a pivotal phenomenon. This conversion is fundamental to the functionality of brake systems, wherein the polymeric brake friction composite interacts with the rotating disc or rotor, thereby absorbing substantial shear and thermal loads [1,2]. For an automotive brake system to be deemed efficient, the friction composite employed must exhibit a plethora of critical attributes. Among these, the main ones are the ability to endure elevated temperatures, sustain a consistent coefficient of friction (CoF), manifest minimal wear and ensure an optimal pedal feel. Achieving this delicate balance is a formidable challenge that has spurred extensive research efforts across the field.

Researchers have extensively studied brake friction composites, exploring various ingredients and formulations to meet demanding performance criteria. However, achieving the ideal balance of fibres, fillers, binders and abrasives to improve tribological performance remains a challenging and ongoing pursuit. A notable study by Ostermeyer, using a full-scale inertia dynamometer, highlighted the extreme conditions brake systems face during operation. It showed that flash temperatures at the pad-rotor interface could reach 1200 °C, with frictional power between 400 – 600 W/cm^2, leading to thermo-chemical reactions that create a durable tribofilm, crucial for maintaining brake system integrity [3,4].

The formation of the third body layer, a significant aspect of the tribofilm, serves a critical function in maintaining consistent friction and wear characteristics [5]. The role of metal sulphides in this context cannot be overstated, as they are pivotal in the establishment and maintenance of the tribofilm. Sathickbasha et al. [6] conducted a comprehensive study on the tribological performance of steel family fibres and the role of individual metal sulphides within standard friction material formulations. Their findings underscored the dual role of metal sulphides as friction stabilisers, highlighting their importance in maintaining consistent performance. Further investigations by Sathickbasha et al. [7] delved into the frictional and wear performance of multiple metal sulphide mixtures.

Potassium titanate fibres (K_2TiO_3) are inorganic compounds known for their excellent thermal stability, mechanical strength and resistance to high temperatures, making them ideal for brake friction composites. Potassium titanate (KT) fibres reinforce the composite, improving wear resistance and maintaining structural integrity at elevated temperatures. Mahale et al. [8,9] investigated adding KT whiskers to a standard friction material and found a 9 % improvement in wear performance. This result shows the value of KT as an ingredient in brake friction composites and the need to explore various materials to improve brake system performance. Chavez Jara and Jang [10] further explored the potential of KT fibres by assessing their performance as platelets and whiskers reinforced with aramid pulp. Their research showed that KT fibres effectively form a third body layer, crucial for maintaining stable friction and wear performance, a similar finding to Mahale et al [9]. Kim et al. [11] suggested combining KT fibres with aramid pulp, highlighting that this blend helps maintain consistent friction. These studies emphasise the potential of potassium titanate fibres to improve brake friction material performance.

The incorporation of nonmetallic ingredients, particularly KT fibres, within brake friction materials is a promising avenue for enhancing performance. The unique properties of KT fibres, including their high thermal stability and mechanical strength, make them an ideal candidate for use in high-performance brake systems. When combined with metal sulphides, which act as friction stabilisers and contribute to the formation of the tribofilm, the synergistic effects can lead to significant improvements in the overall performance of the brake friction material.

The present study aims to advance the understanding of the synergistic interactions between KT fibres and metal sulphides within brake friction materials. The tribological performance of the formulated brake friction material was tested according to the SAE J661 standard, which provides a comprehensive framework for evaluating friction materials. Physical, chemical and mechanical properties were also assessed following IS 2742 (Part 4) guidelines. To understand the wear mechanisms, worn samples were analysed using advanced techniques like scanning electron microscopy (SEM) for detailed imaging of wear patterns and energy-dispersive X-ray spectroscopy (EDS) for elemental analysis.

2. Research methodology

2.1 Materials and formulation

The formulation of the brake friction composites is shown in Table 1. Three formulations [4,6] were prepared by varying the wt. % of potassium titanate (KT) fibre compensating with the lubricant mixture as shown in Table 1. The SEM micrograph of the KT fibre is shown in Figure 1. The fillers consist of barium sulphide (35 wt. %), vermiculite (10 wt. %), cashew dust (3 wt. %) and mica (3 wt. %) which is used to fill the volume occupancy and reduce the overall cost of the fabricated brake pad. A binder of 12 wt. % is considered for the study. The fillers and binders wt. % were kept constant in all the formulations. The wt. % of KT fibre varies from 10 to 30 wt. % and is compensated with the lubricant mixture ranging from 27 to 7 wt. %. The lubricant mixture ingredients and their wt. % are given in Table 2.

Table 1. Brake friction composite ingredients

Ingredient	BS1	BS2	BS3
	wt. %		
Potassium titanate fibre	10	20	30
Fillers	51		
Lubricant mixture	27	17	7
Phenolic binder	12		

Table 2. Lubricant mixture ingredients

Ingredient	BS1	BS2	BS3
	wt. %		
Graphite	10	5	3
MoS_2	10	5	2
Bi_2S_3	5	5	1
Sb_2O_3	2	2	1

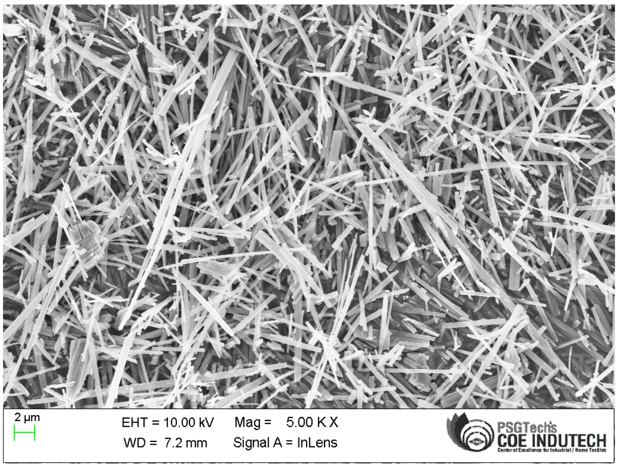

Figure 1. SEM Image of KT fibre

2.2 Fabrication of composites

The brake friction composites are fabricated using the compression moulding technique. In preparing the brake friction composites for the brake pads, the ingredients were mixed using a high-speed shear mixer to ensure a homogeneous distribution of the materials. The mixer operated at a rotational speed of 1500 rpm, which provided sufficient shear force to evenly disperse the fibres, fillers and lubricants. Finally, the binder (phenolic resin in powder form) is added. The mixing process was carried out over 10 minutes to allow complete integration of all components. This was conducted under controlled room temperature conditions to prevent any premature reactions or material degradation [12].

A total of 100 grams of the prepared composite mixture was used to fill the single cavity mould, ensuring complete coverage and uniform distribution of the material. An applied pressure of 17 MPa was maintained in the die cavity to prepare the preform using a hydraulic press as shown in Figure 2. Tata Indica is a model of an automobile, specifically used as the reference vehicle for selecting the back plate used in the brake pad fabrication process.

Figure 2. Hydraulic press with brake pad die: 1 – temperature control knob and display; 2 – hydraulic pressure gauge; 3 – heating coil; 4 – brake pad die

Later, the preform was subjected to curing at 156 °C and applied pressure of 15 MPa for 6 minutes. After curing, the fabricated pad was subjected to a post-curing process in a hot air oven. In this process, the pad was first heated at 145 °C for 1 hour, followed by an additional hour at 150 °C. This gradual increase in temperature allowed the binder to flow evenly throughout the composite material, ensuring uniform distribution and bonding [12,13]. The fabricated samples are shown in Figure 3.

2.3 Characterisation of composites

The fabricated brake friction composites were tested for physical and mechanical properties. The average value of the three sets of samples was considered for the entire study and listed in Table 3. The density of BS3 is higher when compared to other samples (BS1 and BS2). A similar trend is observed in the hardness values and the opposite in the shear strength values of the samples.

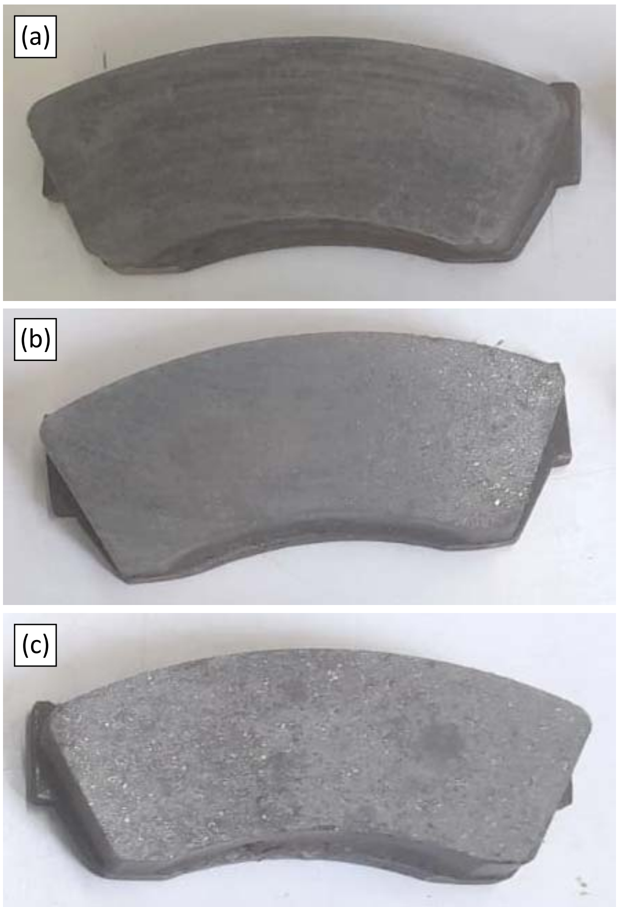

Figure 3. Fabricated samples: (a) BS1, (b) BS2 and (c) BS3

Table 3. Properties of the brake friction composites

Sample	Density, g/cm^3	Hardness (HRL)	Shear strength, MPa
BS1	2.03 ± 0.012	83 ± 3.21	3.24 ± 0.15
BS2	2.04 ± 0.020	83 ± 2.85	3.24 ± 0.15
BS3	2.08 ± 0.018	86 ± 2.80	2.94 ± 0.13

Density was measured using the Archimedes principle, and hardness using a Rockwell hardness tester (HRL scale) where a steel ball intender of 6.35 mm and applied load of 60 kg for the exposure time of 15 s at room temperature is considered [13,14]. The shear strength is calculated using a standard die setup following ISO 6312 standard. Each property was measured three times and the average value is reported in Table 3.

2.4 Tribological testing of composites

The tribological test was carried out using a chase-type tribotest ring following the SAE J661 test schedule as shown in Table 4. To evaluate tribological performance, the sample was cut from the fabricated pad of size 25 × 25 × 8 mm [4,6,7]. The tribological tests were conducted at a constant speed of 411 rpm, with an applied load of 657 N against a cast iron drum. The sample is kept at a distance of 140 mm from the drum centre. The testing temperature varied according to the SAE J661 schedule, ranging from 82 to 345 °C during fade and recovery tests. Each tribological test was repeated three times to ensure reproducibility. Figure 4 shows the chase-type tribotesting machine.

Table 4. Test schedule SAE J661

No.	Stage	No. of applied brakes	Rotor temperature, °C
1	burnish	1	–
2	baseline I	20	82
3	fade I	1	82 – 289
4	recovery I	1	261 – 93
5	wear	100	88
6	fade II	1	82 – 345
7	recovery II	1	317 – 72
8	baseline II	20	88

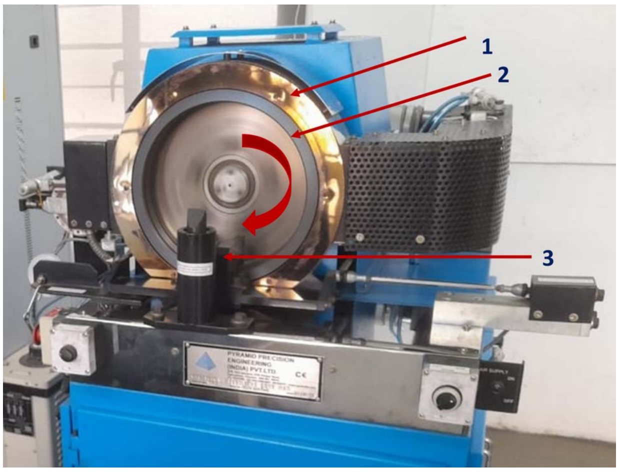

Figure 4. Chase-type tribotest rig: 1 – heating coil; 2 – rotor (drum); 3 – sample holder

The test starts with burnishing, where conformal contact with the sample and cast iron rotor is established. The fade I and II stages were conducted by gradually increasing the temperature from 82 to 345 °C, while the recovery I and II stages involved a gradual reduction in temperature from 317 to 72 °C. These stages simulate real-world braking conditions where the brake system heats up during prolonged use (fade) and cools down during subsequent periods of less use (recovery). The wear performance is evaluated at a constant speed, load and temperature where 100 brake cycles are applied.

3. Results and discussion

3.1 Friction performance

The friction behaviour of brake pad materials during fade and recovery cycles is clearly illustrated by the trends observed in Figures 5 and 6. These graphs demonstrate the fade and recovery performance of different brake pad samples (BS1, BS2 and BS3) under varying temperature conditions.

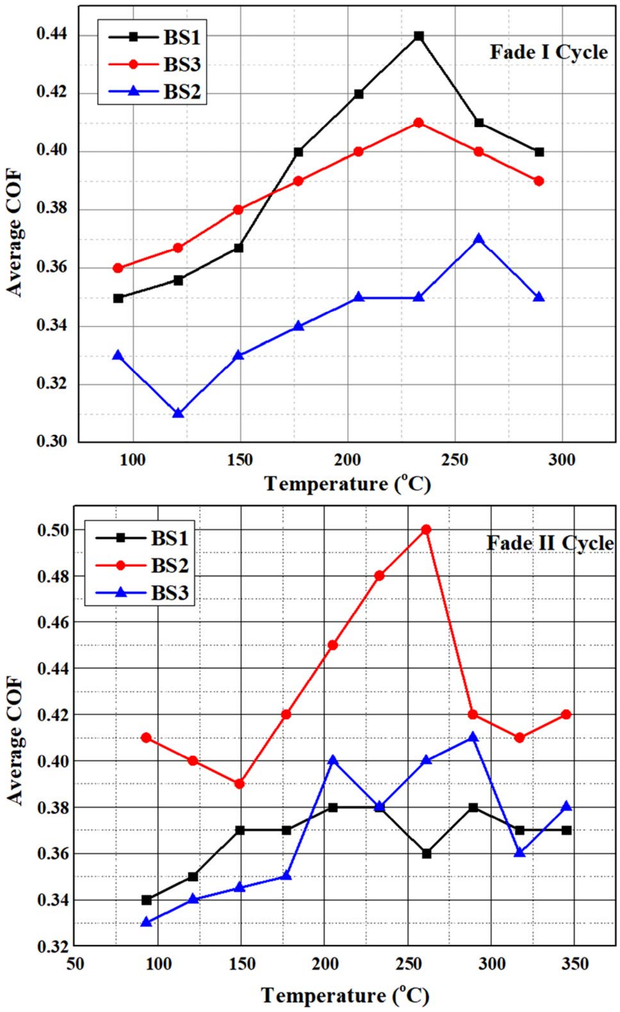

Figure 5. Fade I and fade II performance

For sample BS1 during the fade I cycle, the CoF initially increases from 0.35 to 0.36 within a temperature range of 150 °C and further rises to 0.44 at 230 °C. This trend can be attributed to the adhesive component of friction, where increased temperature enhances the adhesion between the brake pad and rotor surfaces, increasing the CoF. However, as the temperature continues to rise, the CoF declines to 0.40, suggesting that the adhesive bonds may weaken or break due to thermal degradation. This decline can also be related to the abrasive component of friction mechanisms, where the initial increase is due to the hard asperities on the brake pad surface, but these asperities may wear or soften at higher temperatures, reducing the CoF. Additionally, an oxidative component of friction could play a role, where initial oxide layer formation increases friction, but further thickening or uneven distribution of oxides can decrease the CoF [4,7,15,16].

In contrast, sample BS2 shows a gradual increase in CoF with temperature until it peaks at 230 °C, followed by a slight decrease. This suggests a more consistent frictional response, likely due to a well-balanced composition of lubricants and reinforcing fibres. Metal sulphides in the lubricant mixture can form a consistent protective layer, reducing metal-to-metal contact and maintaining a stable CoF. However, in the fade II cycle, BS2 exhibits considerable fluctuations in CoF, ranging from 0.39 to 0.50, before stabilising at 0.42 over a 150 °C temperature range. These fluctuations may be due to variations in the formation and stability of protective oxide layers at different temperatures, indicating challenges in maintaining consistent frictional performance under cyclic thermal conditions.

Sample BS3 exhibits a similar trend to BS2 in the fade I cycle, with its CoF peaking at 0.37 at 270 °C before declining. This indicates difficulty in maintaining a stable CoF at higher temperatures, similar to BS2, likely due to the same mechanisms of adhesive, abrasive and oxidative components of friction. In the fade II cycle, BS3's CoF generally declines and stabilises after reaching 270 °C, showing a pattern similar to BS1, which has fluctuating CoF throughout the cycle. These fluctuations in CoF for samples BS1 and BS3 highlight potential risks in braking performance, as inconsistent frictional behaviour can lead to safety concerns [17,18].

The recovery performance of the brake pad materials, as shown in Figure 6, provides further insights into their ability to regain frictional properties as temperatures decrease. Sample BS2 demonstrates superior recovery performance in both recovery I and II cycles, indicating robust thermal stability and effective frictional property retention. The lubricant mixture and KT fibres likely help maintain CoF by forming protective layers and retaining structural integrity during thermal cycling. In contrast, samples BS1 and BS3 exhibit fluctuations in CoF during the recovery process, suggesting inconsistent material stability and potential issues under varying thermal conditions.

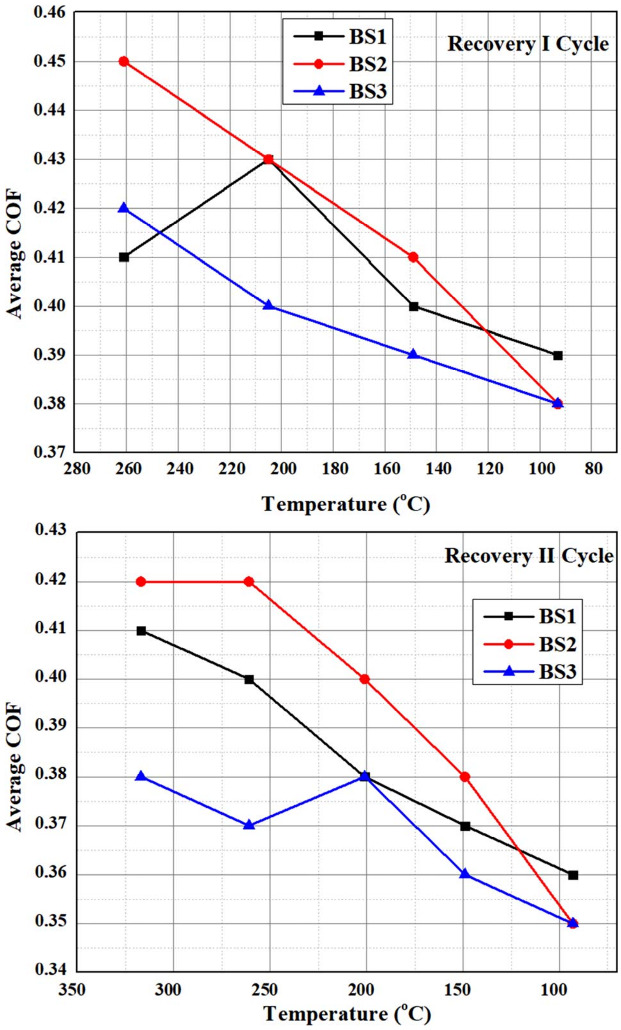

Figure 6. Recovery I and recovery II performance

Several friction mechanisms are evident in the recovery cycles. As temperatures decrease, thermal decomposition and re-formation of material play a significant role in friction recovery. For BS2, the ability to form stable oxide layers and consistent transfer films between the brake pad and rotor enhances frictional performance. In contrast, the fluctuations in BS1 and BS3 may be due to disrupted film transfer and inconsistent oxide formation. The ability of the materials to undergo elastic and plastic deformation during braking and then recover also influences their frictional properties, with BS2 likely exhibiting better recovery due to its material composition. The trends observed in Figures 5 and 6 underscore the importance of having a balanced composition of lubricants, reinforcing fibres and other components to achieve consistent frictional performance. Sample BS2 stands out for its superior recovery and more consistent frictional behaviour, while samples BS1 and BS3 exhibit fluctuations that could compromise safety and performance.

These findings highlight the critical role of material science in designing effective brake pad composites that can withstand the demanding conditions of braking applications.

3.2 Wear performance

The wear behaviour of brake pad materials is critical for their performance and longevity. Figure 7 illustrates the wear performance of three brake pad samples (BS1, BS2 and BS3), measured in terms of % mass loss and % thickness loss (as per industrial standards) [19,20]. Figure 8 gives the thickness loss in millimetres and mass loss in grams. These metrics provide insights into the material's durability and resistance to wear under operational conditions. The study majorly considered the % mass loss and % thickness loss as recommended in industrial practice.

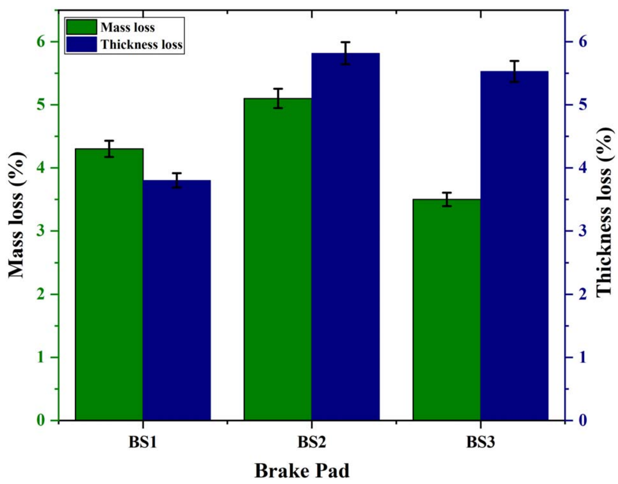

Figure 7. Mass loss (%) and thickness loss (%)

For sample BS1, the mass loss is around 4 %, while the thickness loss is slightly lower, close to 3.5 %. This indicates a moderate level of wear, suggesting that the material has a balanced resistance to both mass and thickness degradation. The wear mechanisms at play here include abrasive wear, where hard asperities on the brake pad surface scrape against the rotor, causing material loss [19]. The moderate thickness loss also points to adhesive wear, where the frictional heat causes localised welding and tearing of the contact surfaces, leading to material removal.

Sample BS2 exhibits the highest wear among the three samples, with mass loss and thickness loss both exceeding 5 %. This significant wear indicates that BS2 is less resistant to operational stresses compared to BS1 and BS3. The primary wear mechanisms responsible for this high wear rate likely include severe abrasive wear, where the harder components of the brake pad aggressively wear the material. Additionally, oxidative wear could be contributing to the high mass loss, where high temperatures during braking lead to the formation of oxides that are then worn, further contributing to mass loss. The high thickness loss suggests that the material's structural integrity is compromised under thermal cycling, leading to more pronounced wear.

Sample BS3 shows a different trend, with a mass loss of around 3 % and a thickness loss significantly higher at approximately 5 %. This discrepancy suggests that while the overall mass loss is lower, the material experiences significant thinning, indicating the existence of adhesive wear. Beyond adhesive wear, thinning vs. mass loss discrepancies can also be attributed to other factors such as oxidative wear and thermal fatigue. During high-temperature conditions, the formation of oxide layers can contribute to material thinning without significant mass loss. This occurs because the oxide layer, while thin, reduces the overall structural integrity, leading to a more pronounced thickness reduction. Moreover, thermal cycling can induce microcracks that further contribute to thinning without corresponding mass loss.

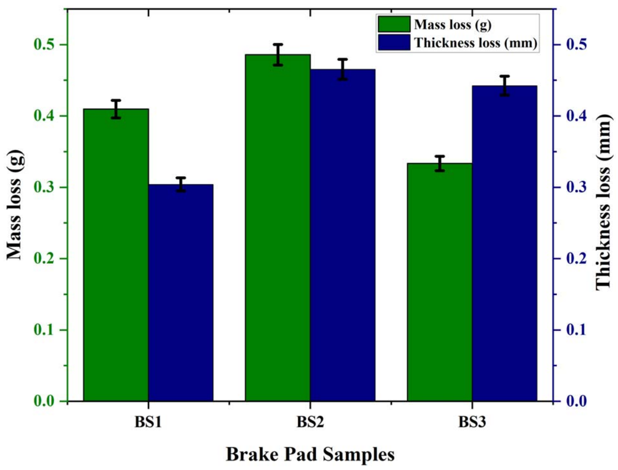

Figure 8. Mass loss (g) and thickness loss (mm)

The influence of density plays a critical role in the wear performance of brake pads. Higher-density materials, like BS3, experienced less mass loss even with considerable thickness reduction. This may be attributed to the material being more compact and less prone to mass shedding during the test. On the other hand, lower-density materials, such as BS1, exhibited higher mass loss due to their less dense structure, making them more susceptible to material removal. Therefore, the influence of material density and the different wear mechanisms acting on the brake pads led to a lack of direct correlation between mass and thickness loss in the brake pads.

The comparative analysis of these samples highlights the importance of material composition and the synergistic effects of lubricants and reinforcing fibres in mitigating wear. For example, potassium titanate (KT) fibres in the brake pad composition can enhance resistance to abrasive wear by providing a hard, stable structure that resists deformation and material removal. Similarly, the inclusion of lubricants such as metal sulphides helps form protective layers that reduce direct metal-to-metal contact, thereby minimising adhesive wear.

The wear performance of brake pad materials is influenced by a combination of abrasive, adhesive and oxidative wear mechanisms. Sample BS1 exhibits moderate wear, suggesting balanced resistance to these mechanisms. Sample BS2, with the highest wear, indicates susceptibility to severe abrasive and oxidative wear, while sample BS3 shows significant adhesive wear. These findings underscore the necessity of optimising material composition to enhance wear resistance, ensuring the brake pad's durability and effectiveness under varying operational conditions.

3.3 Worn surface analysis

The worn surfaces of the tribotested samples BS1, BS2 and BS3 were meticulously characterised using scanning electron microscopy (SEM) coupled with energy-dispersive X-ray spectroscopy (EDS), as depicted in Figure 9. The SEM images across all samples revealed the formation of primary plateaus (marked as A), which are critical in understanding the wear mechanisms and material behaviour under frictional conditions.

For sample BS2, the SEM analysis shows a significantly larger area of primary plateaus compared to BS1 and BS3. This extensive formation can be attributed to the optimal combination of reinforcing fibres and lubricants, enhancing the wear resistance and maintaining a stable coefficient of friction [20,21]. The presence of a rich third-body formation on BS2's surface corroborates its superior frictional performance observed in tests. The EDS analysis of BS2 further reveals a diverse elemental composition, indicating a well-distributed and effective material blend that contributes to its durability.

Sample BS1, although exhibiting a substantial formation of secondary plateaus (marked as C) and moderate primary plateau development, struggled

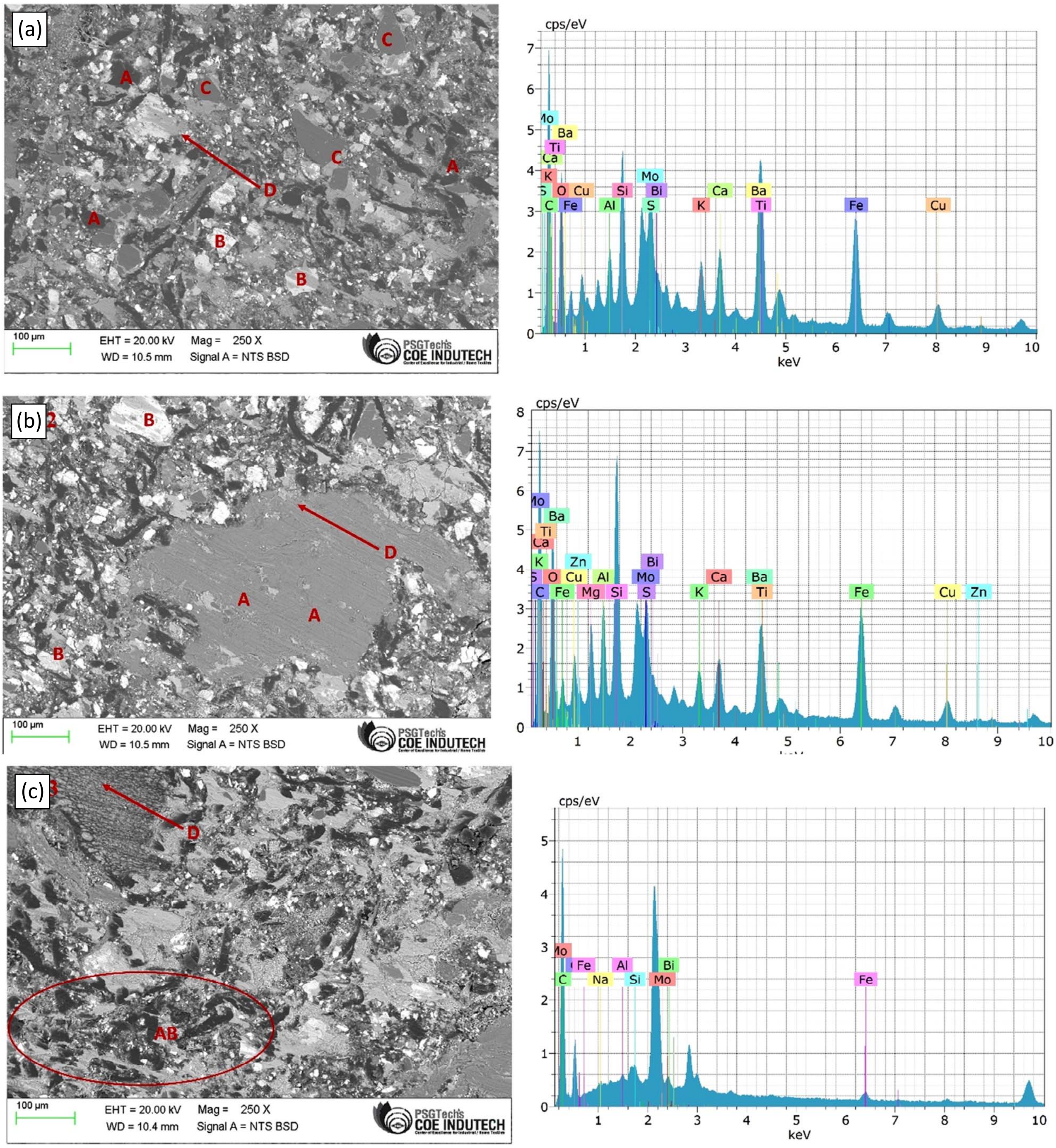

Figure 9. SEM image and EDS analysis of the samples: (a) BS1, (b) BS2 and (c) BS3; A – primary plateaus, B – barytes, C – secondary plateaus, D – direction of rotation, AB – cluster of primary plateaus and wear fragments

to maintain consistent friction during the fade II cycle. This inconsistency is evidenced by the presence of wear fragments on the contacting surface. The EDS analysis of BS1 indicates the presence of various elements, similar to BS2, but the distribution and synergism of these components appear less optimal, resulting in reduced wear resistance and stability.

The SEM images of sample BS3 highlight a different wear mechanism. The surface shows significant mass loss and the formation of micropits, indicative of abrasive wear. The EDS analysis of BS3 reveals a high content of potassium titanate fibres, which, despite their reinforcing properties, seem to contribute to poor third-body formation.

The excessive amount of these fibres may lead to a brittle structure, prone to fragmentation under stress, as evidenced by the cluster of small primary plateaus (marked as AB) and fine asperities. This brittleness and poor third-body formation pose a serious risk of catastrophic failure in friction material applications. The directional arrows (marked as D) on the SEM images indicate the direction of rotation during testing, providing further insights into the wear.

The optimal combination of fibres and lubricants in BS2 resulted in superior primary plateau formation and third-body generation, enhancing its wear resistance. Conversely, BS1, despite moderate plateau formation, exhibited wear fragments due to suboptimal material synergy. BS3's high fibre content led to brittle behaviour and poor third-body formation, significantly affecting its wear performance.

4. Conclusions

The three samples BS1, BS2 and BS3 were fabricated using potassium titanate (KT) fibre and a lubricant mixture (graphite + MoS_2 + Bi_2S_3 + Sb_2O_3). The tribological performance of these samples was evaluated, leading to the following conclusions.

The sample BS3 exhibited higher hardness and density values compared to samples BS1 and BS2. This can be attributed to the higher content of reinforcing fibres, although it also resulted in brittleness and poor third-body formation.

The fade and recovery performance of sample BS2 was superior to that of BS1 and BS3. This indicates better thermal stability and frictional performance, likely due to the optimal synergy between the fibres and lubricant mixture.

All samples showed evidence of primary and secondary plateau formation, which is critical for maintaining consistent friction. The synergism between KT fibre and the lubricant mixture was effective in achieving this.

Sample BS2 had the highest mass loss at 5 %, indicating significant wear compared to the other samples. This suggests that while BS2 performed well in terms of friction stability, its wear resistance needs further improvement.

The study can be extended to explore the synergistic effects of KT fibre and the lubricant mixture in a standard OE (original equipment) formulation under fade III conditions. Further, mathematical modelling of material composition and the temperature against the tribological behaviour of the brake friction composites will add more insight to the existing literature.

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