SYNERGISTIC EFFECT OF ORGANIC- AND CERAMIC-BASED INGREDIENTS ON THE TRIBOLOGICAL CHARACTERISTICS OF BRAKE FRICTION MATERIALS

SINERGISTIČEN VPLIV SESTAVIN Z ORGANSKO IN KERAMIČNO OSNOVO NA TRIBOLOŠKE ZNAČILNOSTI MATERIALOV ZA TORNE ZAVORE

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In this study, the composition of a brake friction material was experimentally investigated with respect to the effects of the proportions of organic (cashew dust) and ceramic ($ZrSiO_4$ and Fe_2O_3) based ingredients on the tribological properties. The tribological properties of the friction materials were evaluated using a Chase-type friction tester. The effect of the ingredient proportions on the wear resistance and friction stability were obtained in relation to the test temperature and the number of brakings. A scanning electron microscope was used to study the effect of braking on the sliding surface of the friction material. Results showed that the complementary nature of the organic- and ceramic-based ingredients provided the optimum friction behaviour, such as the coefficient of friction stability and the wear resistance.

Keywords: sliding friction, brakes/clutches, wear testing, electron microscopy

V študiji je bila eksperimentalno preiskovana sestava materiala za torne zavorne obloge, glede na razmerje organskih (prah indijskih oreščkov) in keramičnih sestavin na osnovi $ZrSiO_4$ in Fe_2O_3 ter na tribološke lastnosti. Tribološke lastnosti tornih materialov so bile ocenjene z uporabo preizkuševalca trenja vrste Chase. Vpliv razmerja sestavin na odpornost proti obrabi in stabilnost trenja je bil ugotovljen glede na temperaturo preizkusa in število zaviranj. Vrstični elektronski mikroskop je bil uporabljen za študij učinka zaviranja na torni površini tornega materiala. Rezultati so pokazali, da komplementarna narava sestavin na ogpanski in keramični osnovi, zagotavlja optimalno obnašanje pri trenju kot sta koeficient stabilnega trenja in odpornost na obrabo.

Ključne besede: drsno trenje, zavore/sklopke, preizkušanje obrabe, elektronska mikroskopija

1 INTRODUCTION

In a brake system, the energy input begins as a driver presses the brake pedal, is then mechanically, hydropneumatically, electrically or with a hybrid method transmitted to the other components and ends at the disc/drum brakes. This kinetic energy is mostly distributed as the heat resulting from the friction between the brake friction material and the disc/drum interface.¹ Thus, the design and material characteristics of the friction material, the disc/drum material where friction is generated and energy transformation occurs are important for the brake system. Especially brake friction materials are crucial for the stopping distance and noise propensity of a vehicle.^{2,3}

A commercial friction material generally contains more than ten ingredients including metallic-, organic-, ceramic-, polymeric-based powders or fibre materials in a thermoset polymeric matrix. The understanding of the synergistic interaction between the ingredients has largely relied on hands-on experiences and systematic studies of friction materials for the optimum brake performance.⁴ In particular, hard ingredients used as abrasives in brake friction materials, with a relatively high hardness control the level of the friction force and remove pyrolyzed friction films at the sliding interface.⁵ The amount of abrasive is limited in vehicle brake pads because it does a lot of damage to the disc.⁶ The abrasives used in commercial brake friction materials are generally ceramic-based, in various sizes and forms of oxides and silicates, such as zircon, alumina, quartz, magnesia, etc. The organic ingredients of the friction composites such as resin, cashew dust, aramid pulp, etc., are softer than the ceramic ones and responsible for the fade (a decrease in the braking efficiency or coefficient of friction with an increase in the average temperature of the braking surface) which is an extremely undesirable feature.⁷

From references list, it is seen that only a few studies report about the synergistic effect of the organic- and ceramic-based ingredients in friction materials and their roles in the brake performance.⁸ On the other hand, individual effects of organic- and ceramic-based ingredients were extensively investigated.^{8–11} The purpose of the present investigation is to investigate the organic- and ceramic-based ingredients together, with regard to the tribological characteristics of a brake friction material.

2 EXPERIMENTAL WORK

2.1 Material preparation

The brake friction materials used in this study are non-asbestos organic (NAO) materials and the proportions of the ingredients are given in Table 1, including the average densities of the finished products. Frictionmaterial specimens were manufactured by mixing, hot pressing, and sintering. The ingredients were weighed and mixed in the given proportions in a plough shear mixer for 10 min. Aramid fibbers were added initially, followed by other pulpy materials and finally by powdery materials. The manufacturing parameters were chosen according to the study of Ertan and Yavuz.¹² The mix was moulded at 150 °C under a pressure of 7.5 MPa for 5 min in a steel die. Heat treatment was carried out in a mechanical convection oven at 165 °C for 12 h. The total amount of ZrSiO₄, Fe₂O₃ and cashew dust was not changed and was set as 15 % of mass fractions for all the specimens.

2.2 Friction testing and microstructure analysis

Friction and wear performances were conducted using a Chase-type friction tester (**Figure 1**), according to the national standard of the Society of Automotive Engineers (SAE) J661, determining the friction coefficient, the friction force, the wear loss and the types of wear. Gray cast iron with a 280-mm diameter and a hardness of 210 HB was used as the counterpart. The applied load was exerted on the specimen in the holder with a closed-loop servo system and the maximum hydraulic pressure was 540 N. The speed was held constant at 411 min⁻¹ and controlled by a variable speed drive.

The test procedure consisted of a burnishing stop, the fade and recovery tests. The test procedure began with the baseline-I operation of 20 applications. This was followed by the fade-I test at constant speed and load, where the frictional force was recorded continuously at 28 °C intervals while the drum temperature rose to 289 °C. Then, the drum was cooled to 93 °C and the frictional force was recorded continuously at 56 °C intervals during the recovery-I test. This was followed by the baseline-II, fade-II and recovery-II test, similar to the first one, but with the temperatures going up to 345 °C. The wear test, which consisted of 100 applications, was conducted at the end of the testing.

The weight of the pads for each sample was measured before and after the friction test, and the specific wear was determined with the mass method following the standard of TSE 555 $(1992)^{13}$ and calculated with the following Equation (1):

$$\omega = \frac{1}{2\pi R} \times \frac{1}{f_{\rm m}n} \times \frac{m_1 - m_2}{\rho} \tag{1}$$

where ω is the specific wear rate (cm³/N m), *R* is the distance between the centre of the specimen and the centre of the drum (m), *n* is the number of revolutions of



Figure 1: Chase-type friction tester Slika 1: Preizkuševalna naprava trenja, vrste Chase

Table 1: Ingredients of the friction materials investigated in this work (in mass fractions, w/%) **Tabela 1:** Vsebnosti tornih materialov, preiskovanih v tem delu (v masnih deležih, w/%)

Ingredients		A1	A2	A3	A12	A13	A23	A123
Ceramic-based abrasives	ZrSiO ₄	10	3	3	6.5	6.5	3	6
	Fe ₂ O ₃	2	9	2	5.5	2	5.5	4
Organic friction modifiers	Cashew dust	3	3	10	3	6.5	6.5	5
Reinforcements		25	25	25	25	25	25	25
Binders		10	10	10	10	10	10	10
Lubricants		20	20	20	20	20	20	20
Fillers		30	30	30	30	30	30	30
Density (g/cm ³)		2.13	2.11	1.80	2.03	2.01	1.95	2.05



Figure 2: Friction test results for A1, A2 and A3 brake-pad materials: a) COF depending on the test temperature (°C), b) the average COF at elevated temperatures and specific-wear rates

Slika 2: Rezultati preizkusov trenja A1, A2 in A3 materialov zavorne ploščice: a) *COF* v odvisnosti od temperature preizkusa (°C), b) povprečje *COF* pri povišanih temperaturah in specifične stopnje obrabe

the rotating disk, m_1 and m_2 are the average weights of a specimen before and after the test (g), ρ is the density of the brake lining (g/cm³) and f_m is the average friction force (N). The densities of the specimens were determined with the Archimedean principle in water, and the density calculations were repeated five times for each specimen after the sintering.

The friction surfaces after the testing were analysed using a scanning electron microscope (Fa. LEO 1455 VP). For all the observations, the samples were carefully cut from an actual-size brake pad in order to avoid any modification of the friction surface, with a sample size of $2 \text{ cm} \times 2 \text{ cm} \times 1 \text{ cm}$.

3 RESULTS AND DISCUSSION

Experimental observations were made to determine the effects of the ceramic and organic constituents on the changes in the *COF* related to the temperature, the average *COF*, the braking number and the specific wear rate. The friction test results for the A1, A2 and A3 specimens are given in **Figure 2**, showing that the *COF* was generally decreased at elevated temperatures. For all the specimens, it is seen that the *COF* continued to increase until 150 °C. The explanation of this behaviour is that the growth of hard particles in the brake material generated a large shear strength and the maximum *COF* at 150 °C.

After this temperature, the *COF* started to decrease. This behaviour can be explained with the destruction of the resin structure and the loss of the local binding properties as well as the formation of the friction film on the surface, called the fade. A compaction of the wear debris generated at the friction interface accounts for the formation of the friction film.^{14,15} After 250 °C the *COF* exhibited a stable change, with the increasing temperature, in the A1 and A3 specimens, but the *COF* stability of the A2 specimen containing a high proportion of Fe₂O₃ was the lowest, especially at elevated temperatures.

The abrasive effect of the Fe_2O_3 powders increased the wear rate (**Figure 2b**) and the wear debris were built at the friction interface and formed a friction film. This friction film reduces the contact between the pad and the disc. This film (with loosened debris, a stable friction level, and low wear rates) can be maintained at various temperatures, as long as it is not destroyed.¹⁶ These results were confirmed with the microstructure analysis given in **Figure 3**. The areas covered with a disconti-



Figure 3: SEM micrographs of the worn surfaces of the brake-pad specimens after the friction test: a) A1-general, b) A1-detailed, c) A2-general, d) A2-detailed, e) A3-general and f) A3-detailed **Slika 3:** SEM-posnetki obrabljene površine vzorcev zavornih ploščic po preizkusu trenja: a) A1-splošno, b) A1-podrobno, c) A2-splošno, d) A2-podrobno, e) A3-splošno in f) A3-podrobno



Figure 4: Friction-test results for A12, A13, A23 and A123 brake-pad materials: a) *COF* depending on the test temperature (°C), b) the average *COF* at elevated temperatures and specific wear rates

Slika 4: Rezultati preizkusov trenja A12, A13, A23 in A123 materialov zavornih ploščic: a) *COF* v odvisnosti od temperature preizkusa (°C), b) povprečni *COF* pri povišanih temperaturah in specifične stopnje obrabe



Figure 5: SEM micrographs of the worn surfaces of the composites in the Chase test machine: a) A12-general, b) A12-detailed, c) A13-general, d) A13-detailed, e) A23-general, f) A23-detailed, g) A123-general, h) A123-detailed

Slika 5: SEM-posnetki obrabljene površine kompozitov v Chase preizkuševalni napravi: a) A12-splošno, b) A12-podrobno, c) A13splošno, d) A13-podrobno, e) A23-splošno, f) A23-podrobno, g) A123-splošno, h) A123-podrobno nuous friction film on the friction surface are shown in Figures 3a and 3b.

The brake pads containing more Fe_2O_3 powders than $ZrSiO_4$ and cashew dust show an unstable friction behaviour and a low fade resistance, and the areas covered with the friction film on the friction surface are increased (**Figures 3c** and **3d**). The surface of brake pad A2 is very rough and exhibits large, locally delaminated friction-film regions. Several reasons may explain these differences, some of which are merely physical, such as the particle-size distribution and the hardness.¹⁷

The average *COF* of A3 at elevated temperatures (between the 165–345 °C) was very high (**Figure 2b**). The abrasive proportion in this brake pad was minimum. The SEM images indicate very thin and locally delaminated friction-film regions on the surface of the specimen (**Figures 3e** and **3f**) that do not reduce the contact between the pad and the disc. As shown in **Figure 2b**, sample A3 exhibited a high wear rate. The increase in the wear rate of sample A3 is associated with the temperature sensitivity of the organic-based cashew dust (**Figure 3e**).

When both the ZrSiO₄ and Fe₂O₃ amounts were increased in a mixture (A12), the average COF at elevated temperatures (between the 165-345 °C) exhibited the lowest value (Figure 4b). The decrease in the COF and a high wear rate (Figure 4), therefore, appeared due to the low fade resistance and the large friction-film regions at the sliding interface (Figures 5a and 5b). The degradation of the organic particles and the increased abrasive effect caused a decrease in the COF at elevated temperatures. When the worn surfaces of A12 were examined, the areas covered with the friction film were larger and thicker than in the cases of the other specimens. The friction-film areas were locally delaminated from each other. These areas decreased the average COF and the stability of the friction material because a friction film reduces the contact between the pad and the disc.¹⁸ The abrasive effect of the ceramic particles caused



Figure 6: *COF* variations related to the number of brakings for the A1, A2, A3, A12, A13, A23, A123 brake-pad materials **Slika 6:** Spreminjanje *COF* glede na število zaviranj pri A1, A2, A3, A12, A13, A23, A123 materialih zavorne ploščice

an increase in the wear rate. Therefore, the maximum wear rate was obtained for sample A12.

The brake pad with a high relative amount of $ZrSiO_4$ and cashew dust (A13) has a stable *COF* at elevated temperatures, but the values are not so high. This result can be explained with the lubricating effect of the cashew dust and the stable friction behaviour of $ZrSiO_4$. The friction-film formation is more homogeneous and locally delaminated than in the case of A12 (**Figures 5c** and **5d**). However, the wear rate is lower than that of A12.

When **Figure 5e** is examined, it is seen that the areas covered with the friction film increased and the stability decreased with the increasing amounts of Fe_2O_3 and cashew dust. The abrasive effect of Fe_2O_3 decreased the stability and eliminated the cashew-dust lubrication effect. The *COF* exhibited an unsteady change, especially at elevated temperatures, as shown in **Figure 4a**. It can be observed that large, locally delaminated friction-film regions formed on the surface of sample A23. A low wear rate with the increased amount of Fe_2O_3 and cashew dust is associated with the strong and durable friction film (**Figures 5e** and **5f**).

Sample A123 exhibited the highest fade resistance among the brake-pad samples due to its stable *COF* changing with high values. When the worn surface of this sample is examined, it can be seen that the friction film was barely formed on the friction interface (**Figure 5g**). The synergistic effect of the ceramic- and organicbased constituents is clearly seen from the wear rate (**Figure 4b**). The wear rate for this sample is the lowest among the tested samples. This optimum tribological behavior of the A123 brake-pad material can be explained with a combination of the stable friction behavior of ZrSiO₄, the aggressive behavior of the high *COF* of Fe₂O₃ and the lubricating effect of the cashew dust.

The *COF* change related to the number of brakings can be seen in **Figure 6**. From the literature review, it is observed that the number of braking applications has the strongest effect on the friction-interface temperature.¹⁹

Consequently, the *COF* change decreases with the number of brakings. The stability of the *COF* is slightly reduced after the 50^{th} braking. It can be seen that A1, A13 and A123 exhibit high and stable *COF*s, while A3, A12 and A23 exhibit low and unstable *COF*s.

4 CONCLUSION

The friction characteristics of the brake pads including organic (cashew dust) and ceramic (ZrSiO4 and Fe₂O₃) based ingredients in different combinations were examined. The results showed that the organic and ceramic ingredients used as abrasive and friction modifiers have strong synergistic effects providing the average COF, the friction stability and the wear resistance. The friction film on the brake-pad material was mainly affected by the proportions of the ceramic-based ingredients. Increasing the cashew-dust proportion increased the average COF in the specimens. The wear resistance of a brake pad was decreased with an increase in the ceramic ingredients. On the other hand, an addition of a certain amount of cashew dust greatly improved the wear resistance. This complementary nature of the organic and ceramic ingredients provided the optimum friction behavior. In this study, the best friction behavior was obtained in the brake-pad material with the proportions of the ingredients close to the composition of specimen A123 (6 % ZrSiO₄, 4 % Fe₂O₃ and 5 % cashew dust).

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