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Tribological screening tests for the selection of raw materials for automotive brake pad formulations

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ABSTRACT

A modified pin-on-disc test was applied to determine tribological properties of typical brake pad constituents. Ball-milling of these ingredients together with iron oxide and graphite provided model materials displaying the main features of real third bodies. Solid lubricants like graphite affected the friction and wear behaviour of Fe_3O_4 powders considerably whereas further addition of hard nanoparticles induced only minor effects. This was corroborated by comparison with modelling results. MoS_2 played a dual role. Depending on special conditions, this ingredient either reduced or increased friction. The latter could be explained, after nanoscopic characterization, by oxidation and destruction of the wear-protecting tribofilm.

1. Introduction

Brake pads are polymer matrix composites usually containing numerous macroscopic as well as microscopic constituents such as filamentary reinforcements, solid lubricants, abrasives and fillers [1]. Technical requirements towards the system are that a constant and stable coefficient of friction (COF) is provided irrespective of environmental conditions such as pressure, sliding velocity, temperature, humidity etc. As discussed by Jacobson's group in Uppsala, part of the requirements are met by the compliant embedding of the so-called primary contact sites in the polymer matrix [2]. A second contribution comes from the formation of a third body layer or tribofilm [1,2]. Why a multi-phase third body at the interface between brake pad and disc is so important in respect to smooth sliding behaviour at a constant COF-level has been shown by previous modelling results [1,3–7]. Since smooth sliding is essential for good brake performance properties, the knowledge of contributions from different pad constituents to the formation of the third body is important. As reviewed e.g. in [1], it has been proved many times by experimental observations that the major component of the third body is iron oxide Fe_3O_4. Thus it was self-evident to start with testing of this ingredient. Fortunately, the tribological behaviour of iron oxides was studied already comprehensively by Kato [8]. We used the same approach and extended the scope to binary magnetite–graphite blends because Fe_3O_4-films with graphite inclusions had been identified to be a major prerequisite for obtaining stable COF-values and smooth sliding conditions [7].

The present contribution considers the tribological behaviour of a number of ingredients, some of which are frequently used for commercial brake pad formulations whereas others are of more academic interest. In addition to tests with the single raw materials we also prepared several binary and ternary blends with magnetite and magnetite–graphite matrices. The latter was done in order to simulate the expected third body structure with iron oxide as major constituent, graphite as essential constituent for providing smooth sliding conditions and one additional ingredient in each case for checking its functionality towards eventually changing the COF-level. In order to achieve the nanocrystalline structure of experimentally observed third bodies [1] we preferred high energy ball milling as the appropriate method for preparing the blends [7]. Powder mixtures with the same volume fractions were also prepared by manual mixing. The latter method simulates a very early stage of third body formation when severe wear may lead to the detachment of ingredient particles from the pad material or spalling of an oxide film from the brake disc.

Without additional heating stage, pin-on-disc tests are not suitable to simulate the tribological behaviour of real braking conditions mainly because in the latter case the high energy input inevitably leads to material modifications at the interface. Our hypothesis is that by using high energy ball milling for preparing

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Table 1
Powder materials and their properties according to suppliers' data sheets.

Raw material	Supplier	Density [g/cm³]	Grain size	Moh's hardness
Magnetite Fe_3O_4-I	Lanxess (Ger.)	4.6	20–200 nm	6
	Morimura Bros./Toda Kogyo (Jpn.)	4.6	220 nm	6
Molybdenum disulphide MoS_2	Rocol (Den.)	5.1	0.5–2 μm	1–1.5
Graphite C	Asbury Carbons (USA)	2.3	10–200 μm	1.5
Silica SiO_2	Sigma Aldrich (Ger.)	2.6	30 nm	7
Potassium titanate $K_2O \cdot 6TiO_2$	Morimura Bros./Toda Kogyo (Jpn.)	1.0	5–7 μm	4
Silicon carbide	SiC	3.2	20–100 nm	9.5
Cashew nut shell liquid derived polymer CNSL-I	Cardolite Corp. (Belg.)	1.1	50–250 μm	~2
Cashew nut shell liquid derived polymer CNSL-II	Cardolite Corp. (Chin.)	1.0	50–250 μm	~2

the powder blends, comparable material modifications as the ones expected under real braking conditions will be induced. Thus pin-on-disc tests with application of ball-milled powder blends may serve as a versatile alternative to full-scale dynamometer tests, at least as a screening method for the selection of promising pad constituents for new pad formulations. From the scientific point of view the tests are suitable for establishing structure property relationships of tribofilms and they may serve as verification tests of sliding simulations based on the movable cellular automata method (MCA) [7].

2. Experimental procedure

2.1. Raw materials

The origins of raw materials together with data obtained by the suppliers are listed in Table 1.

Both iron oxides Fe_3O_4, although provided by different companies, showed similar grain size and physical properties. MoS_2, graphite, potassium titanate and CNSL-particles (polymer particles derived from cashew nut shell liquid) were used in the same size and shape as for commercial brake pad formulations. Silica and silicon carbide powders are commercially available nanoparticles which up to now are not used intentionally for brake pad formulations, but may occur in the third body of ceramic brake discs [9].

2.2. Preparation of nanostructures by mechanical alloying

Ball-milling was performed by Zoz company, as already described in [7,10]. The milling machine was a horizontal style high energy ball mill equipped with water cooling system of type Simolayer® CM01-21 (Zoz GmbH, Wenden, Germany). This machine has a chamber volume of 2 l. A 2000 g ZrO_2 balls (Ø 5 mm) were used at a powder to ball ratio of 1:20. The processing temperature was kept constant at 28 °C. An atmosphere of argon gas was used to prevent MoS_2 from oxidation.

Three levels of kinetic energy were applied by varying the number of rotations per min (rpm): 900, 1150, and 1500; corresponding to line speeds of 5.5, 7.0 and 9.1 m/s, respectively. During step 1, the minor constituents were processed for 5 min at 900 rpm followed by 15 min at 1500 rpm. Subsequently, the magnetite powder was added and milling continued by repeating the sequence 1500 rpm, 45 s followed by 900 rpm, 15 s during 160 min. Thus, total milling time was 180 min. The final step was comprised of discharging by applying 1150 rpm for 10 min, recharging the powder once more, and finally discharging at 1150 rpm for 10 min.

2.3. Preparation of blends by manual mixing

Manual mixing of the raw materials was performed with a ceramic mortar and pestle for about 2 min. Weighing before and after the mixing resulted in loss of about 5–10 vol% of the material due to adhesion at the mortar and pestle.

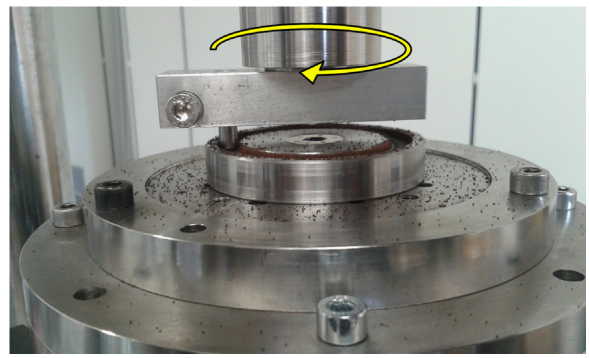

Fig. 1. Photo showing the central part of the pin-on-disc test device.

2.4. Tribological testing

A classical pin-on-disc tribometer of type WAZAU-SST with a maximum applicable load of 2000 N and maximum rotation speed of 3000 min^{-1} was used. Fig. 1 shows a photo of the central part of this device. A rotating pin is pressed against a stationary disc which is fixed on a support. Normal force acts from below the disc by means of a spring and can be manually and accurately applied with a screw that defines the spring's tension.

Both, pin and disc consist of commercial low-carbon structural steel (German grade St52) and were produced by machining of bar stock. As suggested by Kato [8], the disc was 10 mm thick and had an outer diameter of 60 mm with a groove of 6 mm width and a depth of 2 mm in which powder can be supplied which then cannot escape from the contact during sliding. The pin was 17 mm long and 5 mm in diameter with a slight spherical curvature corresponding to a radius of 50 mm and a rounding of the head's edges corresponding to a radius of 1 mm, in order to improve contact with the powder particles and avoid difficulties with misalignment. Furthermore, surface roughness of the pin's head and the groove was adjusted to R_a=0.4 μm.

Two tests were performed with identical powders each. The first was performed with a running-in period of 5–10 min to allow for geometrical adjustment of pin and disc before the powder was supplied. Since wear rate was high during this stage, this also lead to a roughening of the surfaces. The second test was performed without running in period, i.e. the powder was supplied to the grove before the onset of sliding. Usually in that case the original low surface roughness was preserved because there was immediate film formation leading to a considerable reduction of the wear rate, as described in the original paper of Kato [8].

The amount of powder supply was 0.1 g which was found to be sufficient for most tests to form a third body layer within the groove. The majority of tests were performed for 1 h in laboratory