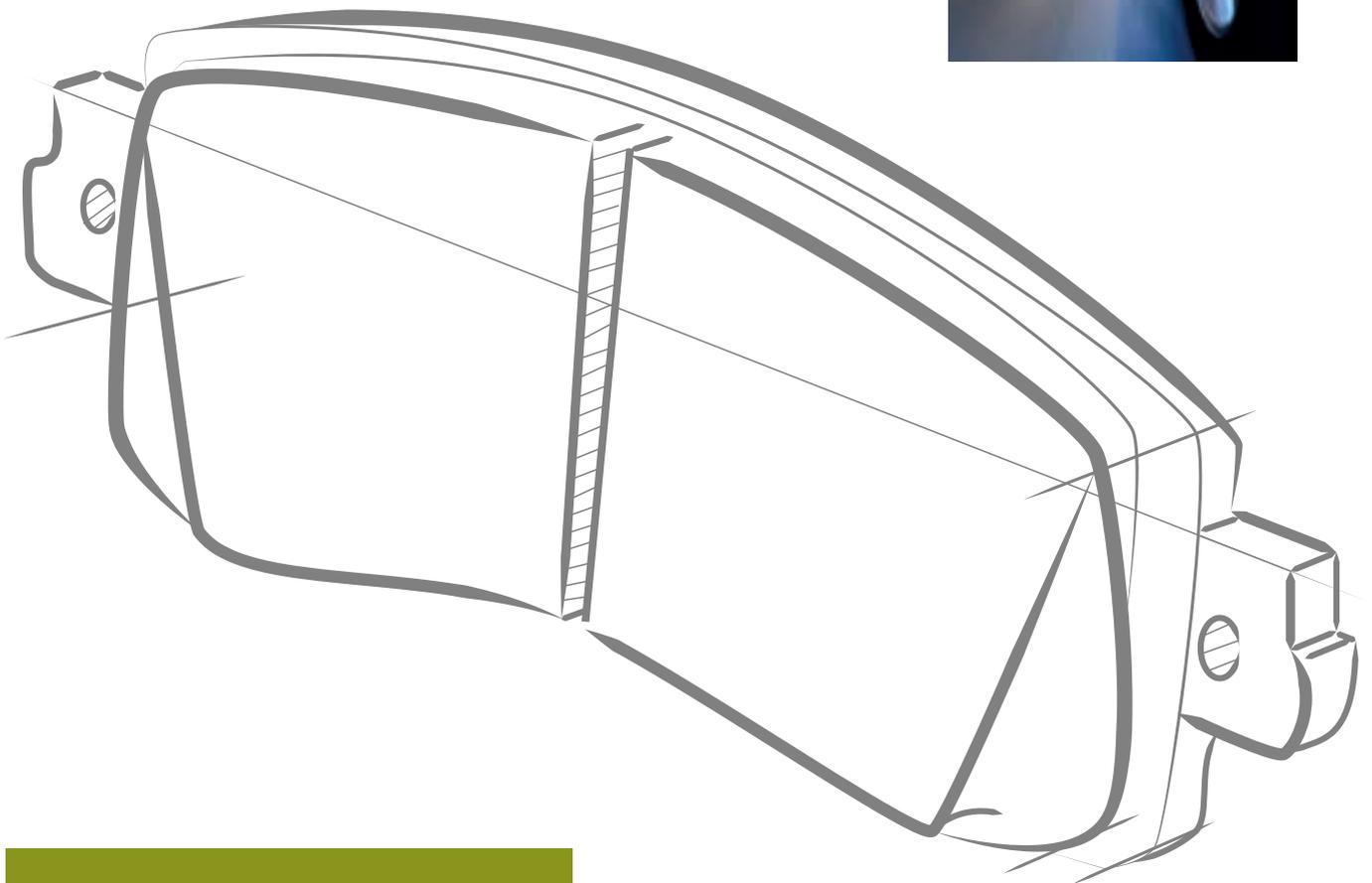


White mineral fibres for reduced wear in friction applications

Technical Paper



Bio-soluble mineral fibres: Alternative chemical compositions and the effect in disc pad applications

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KEYWORDS – engineered mineral fibres, bio-solubility, fibre chemistry, reduce disc wear and dust emissions, copper free.

ABSTRACT

Engineered mineral fibres from Lapinus are used globally in friction applications. They give the friction material a combination of mechanical and tribological properties, in both NAO/non-steel and low steel formulations. Mineral fibres have a pronounced effect on the micrometric scale at the surface of the brake material. They can act as an anchoring point, which effectively promotes the third body layer formation and development. As a result, friction stability and wear resistance are improved. They also influence friction level due to their chemical composition and their contact and interaction with the counterpart, the metallic disc.

With regard to health and safety aspects, these mineral fibres have the important property that they are certified as bio-soluble, which is determined by the specific chemistry. The chemical tolerance range of bio-solubility can be effectively controlled during the production process. Thanks to this, it is possible to offer two different bio-soluble fibre grades to the friction industry.

The fibre chemistry is one of the key characteristics for the unicity of these mineral fibres. The chemical composition has a significant effect on the production process and therefore on the specific physical properties of the mineral fibres. This directly influences friction performance and pad and disc wear.

This paper describes the effect of fibre properties, such as chemical composition and fibre diameter, on friction performance in disc pad applications. The paper shows test results of a new mineral fibre grade, which has been specially developed to offer an alternative performance to current products with the aim of reducing disc wear. This new

product consists of a different chemical composition without compromising the bio-solubility of the fibres. It is shown that overall friction and wear performance are influenced, which opens up new possibilities for friction material formulations.

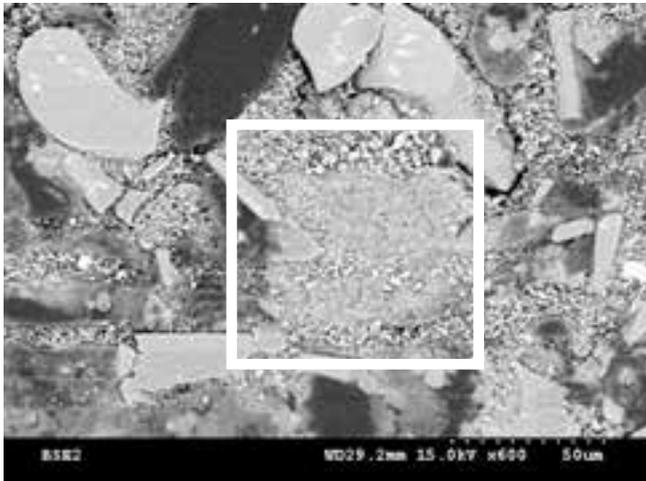
The study shows the friction performance of NAO copper-free materials with the focus on the specific role of the chemical composition of two different mineral fibres at the surface layer of brake pads. With the development of a new fibre grade, it is possible to promote third body layer formation and stabilisation of the friction level, without this having an aggressive effect on the disc surface. With this development it is foreseen that this newly engineered fibre can complement future formulations in reducing wear, and therefore also dust emissions (e.g. wheel dust).

INTRODUCTION

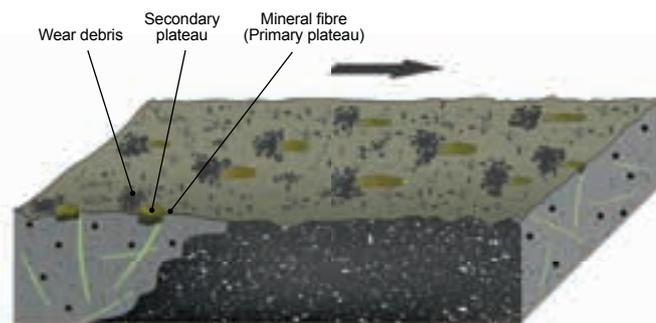
Friction performance is a complex tribological interaction between disc and friction material surfaces. The mechanisms taking place at this contact area during braking are a combination of physical, thermomechanical and chemical phenomena. Kinetic energy has to be converted into heat, plastic deformations, chemical reactions and wear debris (1).

The generated wear debris is responsible for the formation of the so-called third body layer. Depending on the initiation, growth and degradation of this layer the actual friction coefficient and wear are generated (2). To maintain the surface of the friction material effectively attached to the brake pad, anchoring points are needed. Mineral fibres can play a role here as they function as a primary plateau (3). During sliding friction, wear debris can agglomerate around the mineral fibres and start the formation of a secondary plateau.

Figure 1 shows primary and secondary plateaus in the upper layer of the friction material formed thanks to mineral fibres. These anchoring points contribute considerably to improved friction stability and wear resistance (4).



a)



b)

Figure 1: Formation of secondary plateau around mineral fibre anchoring point; a) SEM image of friction material surface after efficiency test showing primary and secondary plateaus; b) Schematic view of plateau formation

The third body layer consists of a semi-continuous layer formed by primary and secondary plateaus and its composition depends on many factors. It is dependent on the components used in the friction material, the quality and composition of the disc, environmental conditions and the braking system in general. Previous investigations (3, 4) have shown that some raw materials promote third body layer formation more than others. Plateaus are usually made up of iron oxide, copper, carbon, silicon and calcium elements. The presence of fibrous anchoring points is proven to be essential in obtaining optimal friction performance aspects, particularly when talking about copper free materials. In current copper-free formulations, the secondary plateaus cannot exist without these structural components in the friction material (5).

MINERAL FIBRE CHARACTERISTICS

Engineered mineral fibres from Lapinus are used all over the world in friction applications, in both NAO/non-steel and low steel formulations. Mineral fibres have an important effect on the micrometric scale at the surface of the brake material. They function as an anchoring point because of their specific shape and alignment in the friction matrix, specific hardness of 6 Mohs and high thermal and chemical stability. Because of these unique characteristics they are able to withstand high friction forces and the heat generated during braking, even if the energy load increases and mechanical reinforcement at the micro surface is necessary. Under these conditions mineral fibres remain stable, whereas other raw materials start to break down (e.g. organic materials) or oxidation reactions occur (e.g. steel fibres). These characteristics are fundamental because friction stability changes when the chemical composition of raw materials is modified. Table 1 shows an overview of the specific fibre properties of existing bio-soluble mineral fibres.

Mineral fibre property	Average value
Fibre length [μm]	125 - 650
Diameter (num. avg.) [μm]	5.5
Non-fibrous material N > 125 μm [%wt]	0 - 30
Specific surface area [m^2/g]	0.20
Hardness [Moh]	6
Melting point [$^{\circ}\text{C}$]	> 1000
Ignition loss [%wt]	< 0.3
Moisture content [%wt]	< 0.1
Specific density [g/cm^3]	2.75

Table 1: Mineral fibre properties

The main fibre parameters that determine friction performance are fibre chemistry, dimensions of length and diameter, purity (shot content), specific surface treatment and coating. Previous research has demonstrated that the correct combination of properties is essential for optimum performance (6) and depends on the field of application, formulation style and customer requirements.

ENGINEERED MINERAL FIBRE PROCESSING

Fibres from Lapinus are certified bio-soluble mineral fibres due to the specific chemical composition. These raw materials are produced using a combination of sustainable volcanic rocks and man-made briquettes with a certified chemical composition. The raw materials are melted in a furnace at a temperature of approximately 1500 $^{\circ}\text{C}$. The homogeneous

melt flows down on a spinning wheel forming long fibres and very small droplets called shot. A specific surface treatment to modify their chemical compatibility to other materials (e.g. binders) may be added during this process. The spun fibres are collected and in a second production step the shot is removed and the fibre length is adjusted under controlled circumstances, resulting in different bio-soluble fibre grades. Bio-solubility is one of the key characteristics for the unicity of these mineral fibres. Figure 2 shows a picture of spun mineral fibres.

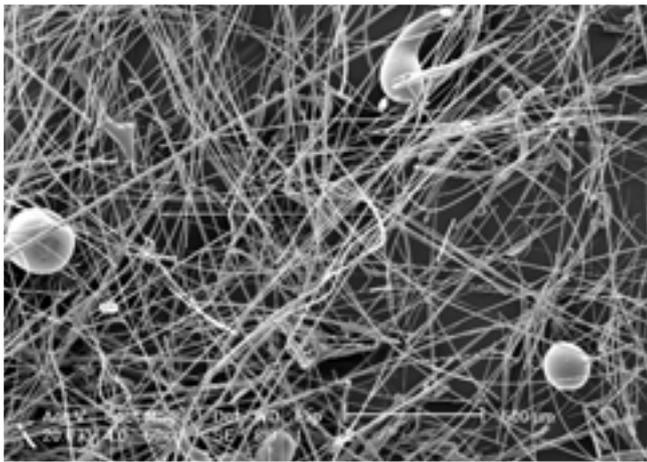


Figure 2: SEM image of mineral fibres before shot removal

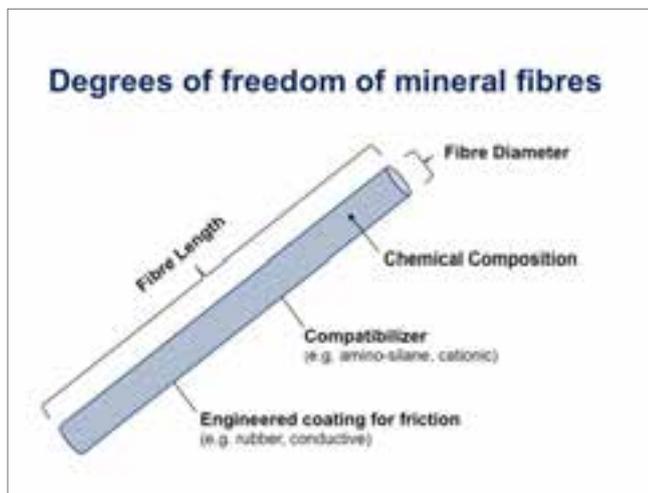


Figure 3: Mineral fibre characteristics

The specific process conditions and quality of the raw materials used are very important for the final physical and chemical product properties of the fibres. Figure 3 shows a schematic view of one mineral fibre and the specific characteristics that are variable and controllable in the production process. The processing conditions have a direct effect on friction performance and can result in higher or lower pad and disc

wear. For this reason the process settings and raw materials are well controlled and kept stable between tight control limits in production. This is essential to ensure constant bio-soluble fibre chemistry within a narrow specification range of bio-solubility.

BIO-SOLUBILITY OF MINERAL FIBRES

The bio-solubility of fibres has been a topic of high concern ever since asbestos was banned in Europe and America. The producing companies realised that Man-Made Vitreous (silicate) Fibres (MMVF) would often be correlated to the physical behaviour of asbestos. Hence large scale epidemiological studies have been started up since the forties and are still running. This strong focus on health and safety and environmental risks has only increased over the years with a peak caused by the Global Harmonization System (GHS), initiated by the United Nations.

If MMVF are respirable, it can potentially cause health problems. The World Health Organization (WHO) established definitions of fibre dimensions in the sixties for the measurement of asbestos fibres. These definitions are currently also used to denote which MMVF are considered to be respirable, being: fibre length (FL) > 5µm, fibre diameter (FD) < 3µm and aspect ratio (FL/FD) > 3 (7). Fibres produced in a spinning process usually meet these dimensions.

If the fibres are respirable, it is important to know if the human body can dissolve and expel the fibres, or if they persist in the lung tissue. This is expressed in the bio-solubility, which is determined by the chemistry of the fibres. Mineral fibres from Lapinus are considered to be respirable. The bio-solubility is proven to be very high and hence the bio-persistency is very low. The fibres are expelled from the body quickly and as such these mineral fibres are considered to be non-carcinogenic. The consistency of the chemistry is kept within a narrow range to ensure the bio-solubility. The chemistry is also controlled by RAL and EUCEB (regular independent sample taking, testing chemistry).

In 1997 the EU directive 97/69/EC classified MMVF with random orientation with alkaline oxide and alkali earth oxide content ($\text{Na}_2\text{O} + \text{K}_2\text{O} + \text{CaO} + \text{MgO} + \text{BaO}$) greater than 18 % by weight as possibly carcinogenic to humans (category 2). Table 2 shows the chemical composition of mineral fibres from Lapinus, confirming that the fibre falls in carc.cat. 2.

Component	Average values high-alumina low-silica mineral fibres [%]
SiO ₂	41
Al ₂ O ₃	20
TiO ₂	2
Fe ₂ O ₃	6
CaO + MgO	25
Na ₂ O + K ₂ O	4
Other oxides	2

Table 2: Chemical composition of mineral fibres

The legislation thereafter provides the opportunity to prove the non-persistence of a category 2 fibre by performing one of the 4 described tests. If the half-time of fibres in lung tissue is under a certain value, the producer can exonerate the fibre from classification. Table 3 shows the half-time of various fibre types in lungs (bio-persistence).

The fibres from Lapinus were tested by the independent institutes Fraunhofer and RCC. These independent measurements confirmed that these mineral fibres are highly bio-soluble and are exonerated from classification.

Fibre type	Weighted half times [T _{1/2}], in days for L > 20µm fibres	
	WT 1/2	95%
Amosite (asbestos fibre)	418	(0-1060)
MMVF32 (E-glass micro fibre)	79	(62-96)
RCF1 (ceramic fibre)	55	(44-66)
MMVF21 (standard stone wool)	67	(56-78)
MMVF33 (475 micro glass fibre)	49	(40-78)
MMVF34 (Lapinus mineral fibre)	6	(5-7)

Table 3: Bio-persistence of man-made vitreous fibres (MMVF) (8)

In 2002 the WHO re-evaluated MMVF using the existing epidemiologic and animal tests as a basis. The conclusion was that the common stone (e.g. MMVF21) and glass wool fibres are not classifiable as carcinogenic to humans (9). Based on thorough animal testing (no epidemiologic tests yet available) of the “newer MMVF” developed in the EU at the end of the nineties (including the MMVF34 from Lapinus, IARC states: “Some of these newer materials have been tested for carcinogenicity and most are found to be non-carcinogenic”

(10). Table 3 clearly shows that in comparison to the common stone wool, the mineral fibre from Lapinus (MMVF34) has a 10 times higher solubility.

Worldwide Lapinus has demonstrated in the various independent studies from different institutes that their engineered mineral fibres are dissolved by the body fluids in a biological way (11) and together with the macrophages (12) the fibres are removed via the respiratory system. The above explanation clearly shows that these fibres are highly bio-soluble and considered to be non-carcinogenic, which is entirely attributed to their unique consistent chemical composition.

The accurate processing method makes it possible to vary the chemical composition in such a way that different bio-soluble fibre grades can be obtained for friction purposes. In addition to the existing bio-soluble fibre grades, a new fibre type has been developed which has a different chemical composition and is also proven bio-soluble. This fibre has a typical white colour determined by the specific fibre chemistry.

This study describes the effect of an alternative chemical fibre composition on friction performance in disc pad applications. The paper presents test results of a newly engineered bio-soluble mineral fibre grade in comparison to existing products and proves the positive contribution of this new fibre.

RESEARCH SET-UP

In order to investigate the impact of fibres with a different chemistry in passenger car disc pads with NAO/non-steel formulations, two different mineral fibres from Lapinus were evaluated and compared with each other: one reference sample of an existing bio-soluble fibre product and one newly developed mineral fibre sample with a different bio-soluble chemical composition. Table 4 shows a summary of mineral fibre samples.

Sample name	Mineral fibre type	Fibre description	Typical property
LF-A	Existing fibre grade	Bio-soluble mineral fibre	Medium fibre length, low shot content
LF-B	New fibre grade	Bio-soluble mineral fibre	Medium fibre length, low shot content

Table 4: Sample names of mineral fibre

Both fibre products were introduced in the same friction material formulation at the same volumetric rate. A more precise description of the NAO/non-steel formulation is given in table 5.

Component	Volume [%]
Novolac resin	16
Aramid fibres	5
Solid lubricants	11
Friction dust/ rubber crumb	10.5
Potassium titanates	17.5
Abrasives	11
Lapinus mineral fibres	5.5
PROMAXON [®] -D	5.5
Fillers	18
Total	100

Table 5: Copper-free NAO/non-steel disc pad formulation

A basic characterisation of mineral fibre products was carried out and differences in fibre properties and fibre chemistry were studied before processing. Macroscopic properties of the friction materials produced were measured and finally friction performances were evaluated (efficiency and wear tests).

Mixing was done in two stages using a multiple blade, high-speed, vertical MTI laboratory mixer (speed 2000 RPM). In the first stage, the aramid fibres and mineral fibres were dispersed by mixing the fibres with graphite and fillers for two minutes. In the second stage, the remaining raw materials were added to the mixture for an additional two minutes of mixing time.

The pads were processed in a positive mould for five minutes under a pressure of 290 kg/cm² at 160 °C. After hot moulding, the pads were cured for four hours at a constant temperature of 210 °C. All the materials were grinded in order to achieve a flat surface and acclimatised for 24 hours at 23 °C and 50% relative humidity afterwards.

INSTRUMENTS AND TESTING PROCEDURES

Fibre length and fibre diameter measurements were performed using a Carl Zeiss Axioskop 2 with an AxioCam digital camera for image processing.

For shot measurements a Hosokawa Alpine 200LS-N air jet sieve was used with sieves of 45, 63 and 125 µm.

The chemical composition of the mineral fibres was determined using a XRF-Axios EP 264 spectrometer for X-ray fluorescence analysis.

Thermogravimetric analysis was carried out in a Mettler TGA/SDTA 851 instrument between 40 and 900 °C at a heating rate of 10 °C/min in air.

Efficiency performance was evaluated using a Horiba dynamometer according to SAE J2522. The pads were scorched, but did not contain an anti-noise shim, slot or chamfer. The brake system used for friction performance and wear testing was the front brake of a VW Golf (WVA21974), using a ventilated disc and inertia of 65 kg.m².

A Krauss machine was used for wear testing as a function of temperature. First all brake pads were analysed according to the ECE R90 annex 8 procedure (constant torque). Temperature block tests were then carried out at 100, 300 and 500 °C adapted from the SAE J2707 wear procedure.

TEST RESULTS

In order to analyse the effect of an alternative chemical composition of mineral fibres, results are divided into two categories: characterisation of fibre material properties and tribological test results. The influence of different fibre chemistry is described in relation to specific fibre dimensions, non-fibrous material (shot) and thermal stability. The tribological test results show the effect of chemistry on the friction performance and wear behaviour of brake pads and disc. The effects shown are the consequence of the specific fibre characteristics which vary between the existing and new bio-soluble fibre grades: samples LF-A and LF-B.

MATERIAL PROPERTIES

To be able to compare fibre characteristics and tribological results of existing bio-soluble fibres and newly developed bio-soluble fibres, the chemical composition of both fibre types is first determined. Table 6 shows the chemical analyses of sample LF-A and LF-B.

Component	Chemical analysis LF-A [%]	Chemical analysis LF-B [%]
SiO ₂	42.7	39.8
Al ₂ O ₃	18.5	19.0
TiO ₂	1.3	0.5
Fe ₂ O ₃	7.7	0.8
CaO	20.6	32.8
MgO	6.0	5.2
Na ₂ O	2.2	1.2
K ₂ O	0.6	0.4
P ₂ O ₅	0.2	0.2
MnO	0.2	0.1

Table 6: Measured chemical composition of mineral fibres

This table clearly shows the difference in fibre chemistry between sample LF-A and LF-B. The new chemistry contains a lower amount of iron oxide and has a higher amount of calcium oxide. Other oxide contents are for both fibre grades in the same range. This new chemical composition also ensures the bio-solubility of the fibres; the values comply with the EUCEB and RAL.

The effect of fibre chemistry on temperature resistance was determined using thermogravimetric analysis. Figure 4 shows the TGA curves measured for sample LF-A and LF-B.

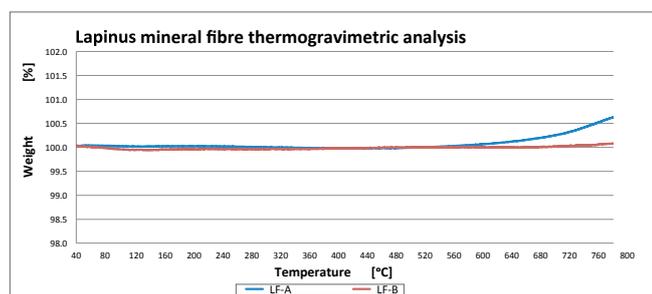


Figure 4: mineral fibre TGA curves, existing versus new fibre grade

Both fibre types are thermally stable up to high temperatures. In a temperature range up to 800 °C the fibres do not decompose. At temperatures above 700 °C they show a slight oxidation behaviour, indicated by a slight increase in weight.

To determine the chemistry effect on fibre dimensions and the amount of non-fibrous material, fibre length and diameter were measured and various shot fractions were analysed. Measurements of sample LF-A and LF-B are shown in table 7. A comparison was made of medium length fibre types.

Fibre property	LF-A	LF-B
Fibre length (avg.) [µm]	306	303
Diameter (num. avg.) [µm]	5.2	4.0
Aspect ratio [FL/FD]	59	76
Shot content > 45µm [%wt]	0.9	1.9
> 63µm [%wt]	0.3	0.9
> 125µm [%wt]	< 0.1	< 0.1
Specific density [g/cm ³]	2.75	2.71
Colour [visual]	Grey/green	Off-white

Table 7: Overview of measured fibre properties for existing and new fibre grade

Because the degree of process control is extremely high and consequently the production conditions are very accurate, it is possible to vary the process settings in such a way that the fibre length of the new fibre grade is similar to the existing bio-soluble fibre product. The fibre diameter of sample LF-B is

smaller as a result of the different chemical composition and the viscosity of the melt. This results in a higher aspect ratio for this new bio-soluble fibre type.

Shot values for the fraction above 125 micrometres are comparable and very low. Sample LF-B has a slightly higher shot amount for smaller shot fractions, but shot values fluctuate slightly over the various production runs and are within the narrow product specification.

TRIBOLOGICAL RESULTS

Table 8 shows a summary of the evaluated friction and material properties influence by the formulation and production process.

Material property	LF-A	LF-B
Density [g/cm ³]	2.26	2.24
Hardness [HRS]	83 stdev 9	80 stdev 6
Porosity [%]	18.7	19.3

Table 8: Macrometric properties

Both samples show similar values for density and porosity. There is no significant difference when using existing or new fibre types.

Hardness measurements were carried out at 10 different points on the friction material surface. There is no significant difference between sample LF-A and LF-B and standard deviations are in the same measurement range. This shows that the new fibre does not affect the hardness of the friction material.

Efficiency and wear tests were carried out with the friction materials produced. Figure 5 shows a comparison between the summary of the AK-Master test results for sample LF-A and LF-B.

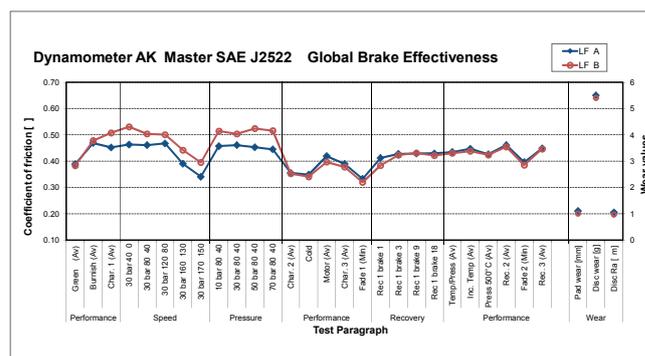


Figure 5: AK-Master efficiency summary comparison

Friction materials containing the new bio-soluble fibres show a higher friction level for speed and pressure sensitivity sections. The other sections of the efficiency test show similar performance levels. The wear values of brake pads and disc are slightly lower for sample LF-B, but not significantly. The results of the new fibres clearly show that a higher friction coefficient does not adversely affect the wear.

Temperature block tests were performed to evaluate the effect of the newly developed bio-soluble fibres on pad and disc wear. Prior to this test, all samples were analysed according to ECE R90 to compare the friction level of the different friction materials and correlate this to wear results.

Figure 6 shows the Krauss ECE R90 Global Spec efficiency results for sample LF-A and LF-B. It clearly demonstrates that the friction coefficient with the new fibre grade is higher in comparison to the existing one. This confirms the results of the AK-Master test and proves again that the new fibres ensure a higher friction level. At the same time sample LF-B showed considerably reduced disc and pad wear during this efficiency test. Pad wear is almost 30 percent lower. Disc wear is approximately 0.3 grams less.

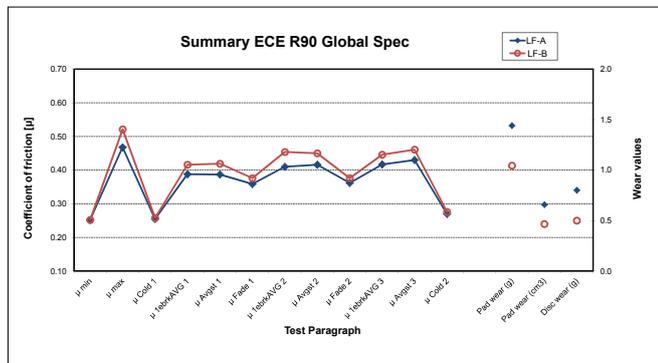


Figure 6: Krauss ECE R90 efficiency summary comparison

To determine the effect of the new fibre properties on wear aspects, extensive wear tests were performed at 100, 300 and 500 °C. Figures 7-8 show the results of pad wear.

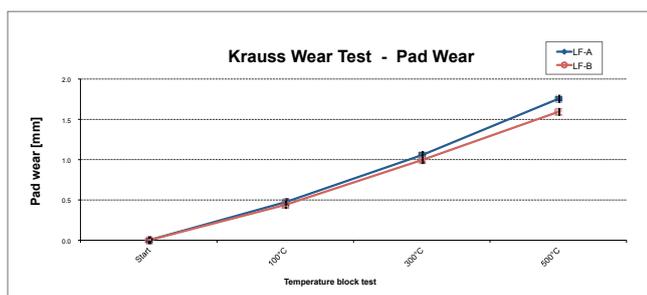


Figure 7: Pad wear comparison after SAE J2707 wear testing



Figure 8: Total pad wear

Pads with the new fibre grade show slightly lower pad wear for medium and high temperature levels. At 300 °C there is only a small difference visible. At 500 °C the results of sample LF-B show a decreasing wear trend. Total pad wear is approximately 10 percent lower when using the new fibres in this specific formulation.

Figures 9-10 show the results of disc wear.

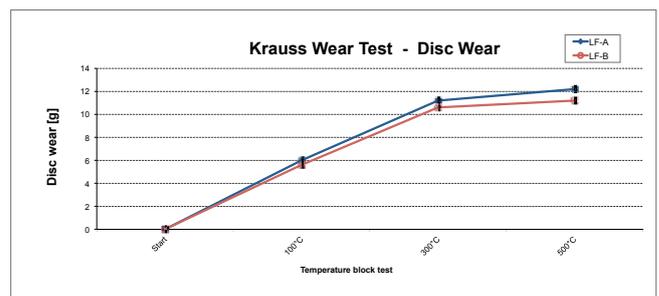


Figure 9: Disc wear comparison after SAE J2707 wear testing



Figure 10: Total disc wear

Here also the new bio-soluble fibre shows an improvement at medium and high temperatures. At 300 °C there is only a small difference in disc wear values. At 500 °C the results of LF-B show a decreasing trend in disc wear. Total disc wear shows an overall reduction of 1 gram when using this new fibre grade.

The temperature block tests show a positive effect of the newly developed bio-soluble fibres on wear performance. Both pad and disc wear are reduced, while the nominal friction coefficient is even higher in comparison to the existing fibres.

DISCUSSION

Test results show that the new mineral fibre grade has a significant contribution to performance and wear aspects in friction materials. The new fibre corresponds to the bio-soluble fibre composition and is less aggressive to the disc surface. The special fibre chemistry contains a lower amount of iron oxide and has a higher amount of calcium oxide, resulting in a less abrasive fibre behaviour. As a result this newly engineered fibre can positively contribute to the lifetime of the brake pad and disc.

Another positive aspect of this special chemical composition is reflected in the specific fibre diameter. Because of the process settings and viscosity of the melt, this chemistry results in fibres with a typically smaller average fibre diameter. This diameter results in a higher fibre aspect ratio (FL/FD) for fibres with similar length (13). A higher fibre aspect ratio improves their mechanical micro reinforcement capacity as has been proven in theory (14).

In addition to this known effect, the smaller diameter results in an increased amount of mineral fibres in the same mixture volume. This leads to more fibres per gram, which theoretically improves reinforcement and may result in the formation of more primary plateaus and as a consequence more secondary plateaus. The promotion of a third body layer will be enhanced and as a consequence this anchoring effect improves friction stability and increases wear resistance. As a result, friction coefficient is increased without compromising wear results.

According to formula 1 and assuming an average fibre length and specific density as described in table 7, it is possible to calculate the amount of fibres in 1 gram.

$$V_{\text{fibre}} = \pi \cdot r^2 \cdot h \quad (1)$$

V_{fibre} = Volume in [cm³]

r = Radius in [cm]

h = Height in [cm]

The number of mineral fibres of the newly developed fibre grade is almost twice as large as the quantity of an existing bio-soluble fibre product; 1 gram of fibre material contains an average of 56 million loose LF-A fibres, but when calculated for LF-B it has 97 million loose fibres. The results clearly show the contribution of an increased number of fibres on friction stability and wear.

When taking into account the fact that the overall friction coefficient is higher for the new fibre grade, the wear results

could even improve and should show larger differences in comparison to commercially available fibre grades, in which case friction levels will become equal. This could easily be achieved by decreasing the amount of abrasives in the formulation or by adjusting the lubrication system. In this case the mineral fibre will provide various functionalities; the fibre will act as an anchoring point and gives micrometric reinforcement at the surface of the friction material, it will promote third body layer formation and will function as adhesive material in the formula.

Overall, the fibres play a major role in the friction matrix. The correct combination of anchoring materials and materials promoting the formation of a third body layer improves friction level, friction stability and wear behaviour of NAO copper-free friction materials for passenger car disc pads. The introduction of this new fibre grade demonstrates the possibility of reducing disc and pad wear, while the friction performance remains at the same level.

CONCLUSION

This study shows that the chemical composition of mineral fibres has a significant influence on the specific physical properties of these anchoring materials. This is directly related to the formation of a third body layer and as a result to friction performance and wear resistance of brake materials.

The newly engineered mineral fibre from Lapinus represents a new alternative chemical composition to current commercial fibres; it is highly bio-soluble and represents no risk for health. This new fibre product enhances the anchoring effect in the upper layer of the friction material. As a consequence the amount of primary and secondary plateaus and thus the actual contact area will increase, which effectively contributes to the promotion of third body layer formation.

The combination of a less abrasive fibre and a smaller specific fibre diameter results in a less aggressive behaviour on the disc surface in comparison to existing mineral fibres. The new fibre increases friction level without compromising wear and can be used in formulations to maintain a stable friction coefficient with the aim of reducing pad and disc wear. The friction coefficient shows improvement for speed and pressure sensitivity sections, while friction stability in general remains unaffected.

It is foreseen that this specially engineered fibre will complement future formulations in reducing wear and therefore also wheel dust and dust emissions.

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