

# Utilization of GDOES for the study of friction layers formed on the surface of brake discs during the friction process

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**Abstract** The paper deals with the chemical characterization of friction layer, generated on the surface of friction materials during the friction test using the glow discharge optical emission spectrometry (GDOES). Friction layer is formed during the friction process and its character depends on several factors. One of the most important factors is the material composition of brake pads and brake disc. When the brake pressure is released at the end of the braking event, the specific friction layer remains on the surface of both pairs (brake pads and brake disc). Using the GDOES profile analysis, the thicknesses of friction layers were estimated and these results were compared with results obtained by scanning electron microscopy together with X-ray microanalysis.

**Keywords** GDOES · SEM-EDX · Friction layer · Friction process · Brake disc · Brake pad

## Introduction

Friction composites are used in several industrial areas and one of them is manufacturing of brake linings for vehicle brake pads. The task of the braking system is to convert kinetic energy of the vehicle during deceleration into

thermal energy. Friction material together with the design of braking system have decisive impact on the proper operation of braking system. The main requirements are adequate value of the friction coefficient and its stability at elevated temperatures, wear resistance and their behaviour related to comfort (noiseless, no vibrations, etc.). The essential requirements are also low production costs and environmental friendliness.

Optical emission spectrometry with glow discharge (GDOES) using the profile analysis is a suitable method for the characterization of the chemical composition of surface layers with high uniformity, e.g., zinc layer on the surface of galvanized sheet (Vontorová et al. 2012).

In this work, GDOES is used to estimate the thicknesses of friction layers. These results will be compared with results obtained by scanning electron microscopy together with X-ray microanalysis (SEM-EDX).

## Theoretical

Tribological and wear process both lead to the creation of friction layer on the surface of friction materials having different tribological properties compared to the original friction materials. Friction contact layer, thus, bears the load, affects the tribological behaviour and the wear. The structure and chemical composition of this layer is significantly different from the composition of the base friction material, but there is not a simple relation between these compositions (Strafelini et al. 2015).

Friction materials used in the automotive industry are considered as some of the most complex composite materials. They may contain even more than 20 different components with variable concentration range. They are designed to maintain a stable friction coefficient, strength,

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wear resistance—all in a wide range of conditions (Chan and Stachowiak 2004; Matějka et al. 2009; Fan et al. 2009; Ma et al. 2008; Matějka et al. 2013).

Brake discs are usually made from cast iron. Basic microstructure of discs contains graphite flakes in a pearlite matrix. Besides the required thermal properties of grey cast iron, cast iron has sufficient mechanical strength, sufficient wear resistance and it is not expensive (Eriksson et al. 2002; Österle and Urban 2006).

The process of friction is influenced by the formation of friction fragments which adhere to the friction surfaces and form a friction layer which is easily visible when comparing the surface of brake pads before and after the dynamometer friction test. Similar layer can be also observed on the surface of cast-iron brake disc (Filip et al. 2002).

Components of friction material (brake pads) exhibit very different wear properties. Steel fibres and abrasive particles have a relatively high wear resistance. The wear is initially caused by separation of particles from a phenolic matrix near large and hard constituents, forming protruding primary platforms. After a short running-in period, the metal fibres forming the central part of the contact area will be located slightly higher than components that are less resistant to wear. This creates nucleation sites. Contact area may grow by a process of agglomeration and compaction of wear particles around the nucleation sites. Particles, or rather, wear debris tend to accumulate near those areas where they are compacted according to the contact pressure. In this manner, secondary areas of small compact particles coming from wear of the pad as well as the disc material are formed. Friction layer at the interface between the brake disc and the brake pad is, thus, composed of two types of contact surfaces—primary and secondary surfaces (Verma et al. 2015).

A thick friction layer is formed mainly in protrusions, filled with friction debris. “Opened areas” are mainly associated with the presence of graphite and vermiculite near the surface. Graphite and vermiculite are known for their lubricating properties; low friction of adhesion layer can, therefore be attributed to that fact (Filip et al. 2002).

Stable friction layer formed on both brake pads and brake disc surface is responsible for final friction-wear performance and its characterization is, thus, of great importance. There are several techniques used for characterization of friction layers while most often used method is scanning electron microscopy. The aim of the paper is to evaluate the suitability of GDOES for characterization of friction layer formed on the surface of brake disc. Unlike the microscopic methods, GDOES has significantly lower detection limits, and therefore, it can quantitatively determine elements with weight concentrations below 0.01% in the surface layer.

## Experimental

Brake discs made of grey cast iron and brake pads based on organic resins were used in this work. Both components are marketed at lower price used for the lower-middle-class cars. Their price ranged from about 10–40 EUR. Two brake discs–brake pads pairs with different chemical composition, and manufactured by different manufacturers are studied in this work.

The chemical composition of the brake discs (Table 1) was determined using the BULK analysis of GDOES method. The chemical composition of the brake pads was determined by X-ray fluorescence spectrometry (Table 1).

### Testing using full-scale brake dynamometer

Testing of friction materials and preparation of friction layer on the brake discs was conducted in the Laboratory of Composite Friction Materials of Centre for Nanotechnology, VSB–Technical University of Ostrava, on the dynamometer Link M2800 (Fig. 1), which is used for testing of friction and wear performance of car and

**Table 1** Chemical composition of the brake discs and pads before friction test

	Brake discs		Brake pads		
	A wt%	B		I wt%	II
C	3.649	2.966	Fe	<b>37.9</b>	<b>65.0</b>
Mn	0.697	1.113	Mn	0.141	0.174
Si	<b>1.05</b>	<b>0.98</b>	Si	<b>2.65</b>	<b>0.55</b>
P	0.05	0.068	P	<0.001	0.009
S	0.065	0.093	S	2.86	0.289
Cr	<b>0.143</b>	<b>0.117</b>	Cr	<b>0.004</b>	<b>0.023</b>
Ni	0.027	0.033	Ni	0.015	0.011
Mo	0.002	0.004	Mo	0.037	0.004
Cu	<b>0.059</b>	<b>0.048</b>	Cu	<b>6.399</b>	<b>0.021</b>
Ti	0.02	0.016	Ti	0.329	0.012
Pb	<0.001	<0.001	Pb	1.064	0.37
Al	<b>0.012</b>	<b>0.013</b>	Zr	0.412	<0.001
Co	<0.001	0.018	Al	<b>0.553</b>	<b>0.753</b>
B	0.005	0.003	Ba	8.303	<0.001
V	<0.001	<0.001	Mg	1.145	0.35
W	<0.000	<0.001	K	0.186	0.068
			Ca	0.326	3.399
			Zn	2.013	0.008
			Sr	0.012	<0.001
			Cd	0.009	<0.0005
			Sn	0.152	<0.001
			Sb	4.438	0.588

Bold values indicate GDOES profile analysis as friction layer indicators



**Fig. 1** Dynamometer link M2800

motorcycle friction materials. Testing took 17 h. 404 breakings were carried out in 18 sections. The initial temperature in majority of the sections was 100 °C; ambient temperature was 20 °C. Load corresponded to the weight of the vehicle for which the brake pairs had been designed (1700 kg), and it was recalculated for the front wheel of the vehicle.

Testing of friction materials has been carried out according to the testing standard SAE J2522 (SAE 2013). This standard defines the test procedure on a dynamometer, which evaluates the effectiveness of friction material with respect to pressure, temperature and speed for vehicles with hydraulic brakes. The main purpose is to compare the friction materials under the same conditions.

Brake pads were weighted before and after the friction test. Weight loss of brake pad I after the friction test was 12.9 g and the weight loss of brake pad II after the friction test was 38.0 g. The big difference in weight loss indicates a different quality of brake pads. It should be emphasized that the products of brake pads wear significantly harm the environment.

### Experimental method

The optical emission spectrometer with glow discharge Spectrum Analytik GmbH (GDA model 750) was used for the characterization of BULK chemical composition of brake discs, and for the characterization of friction layer formed on the surface of brake disc, the optical emission spectrometer with glow discharge Spectrum Analytik GmbH (GDA model 750) was used. BULK analysis was performed to determine the basic chemical composition of the brake discs under the excitation conditions of 700 V and 35 mA. Then the profile analyses were carried out on the brake discs for the characterization of the friction layer under the excitation conditions of 996 V, 15 mA,



**Fig. 2** The brake disc after GDOES profile analysis

2447 hPa. Profile analyses were carried out for 250 s. Brake discs were measured after the friction test as received (Fig. 2).

After dynamometer testing (Fig. 3), the cross-sections of the brake discs were observed with an optical microscope Olympus IX70 and scanning electron microscope, Philips XL30 with energy-dispersive X-ray microanalysis EDX. SEM images were taken by a secondary electron detector at a voltage of 25 kV. EDX analysis of the base material and the friction layer was also performed.

## Results and discussion

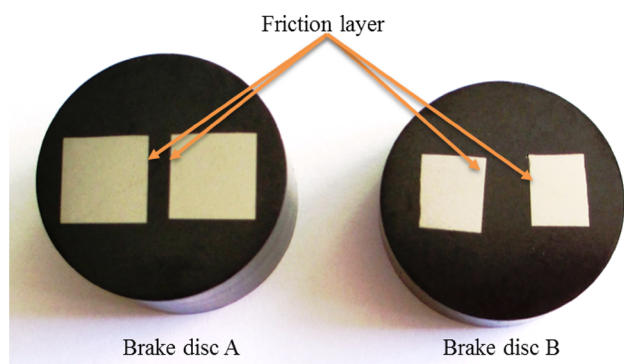
### Choosing the elements characterizing the friction layer

To determine the thickness of the friction layer on the surface of the brake discs, it is important to determine a suitable chemical element which can serve as a marker of the layer. Basic requirements for this element are as follows:

- a constant content in the base material of the brake disc,
- a different content in the friction pairs.

These requirements play an important role with regard to the content of each element in the surface layers of the discs after the friction test. In the following profile analysis of the brake discs, it is verified that the surface layer of the brake disc may be enriched by these elements if their content in the brake pads is higher than their content in the brake disc.

The choice of appropriate elements as markers of the friction layer is based on profile analysis of the disc before the friction test. Only elements whose concentration in profile is constant and whose stabilization time during the



**Fig. 3** Samples of brake discs for SEM

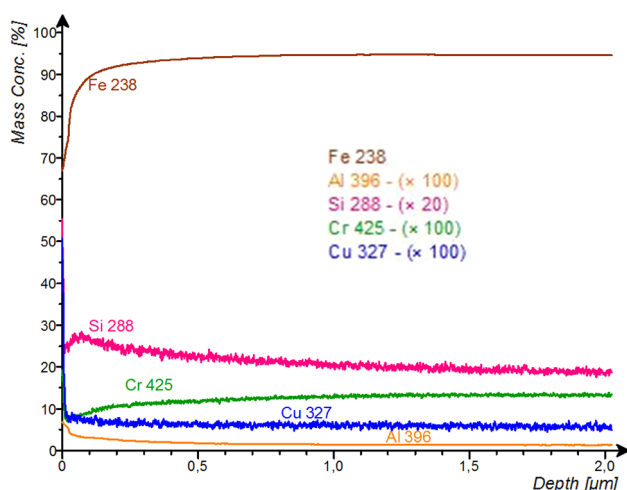
analysis is very short (i.e. in seconds) are suitable. Si, Cr, Cu and Al were selected as markers for the profile analysis (see Fig. 4 as an example for disc A). Other elements were excluded due to long period needed for stabilization of their constant content.

For greater clarity, the Y-axis representing concentration of elements was re-scaled (concentration resulting from the graph should be divided by the number in brackets in the graph legend, e.g. content of Si in Fig. 4 is:  $W_{\text{Si}}(\text{in depth } 2.0 \mu\text{m}) = \frac{21\%}{20} = 1.05\%$ , where  $W$  is mass concentration).

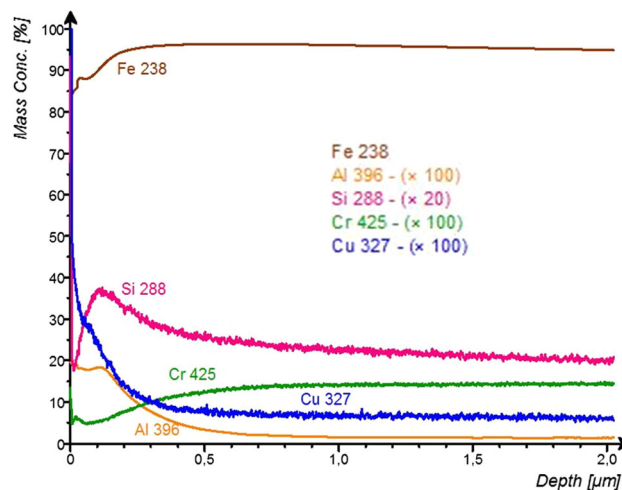
### Chemical composition of friction layers

Figures 5 and 6 show GDOES profile analysis of disc A and B after the friction test.

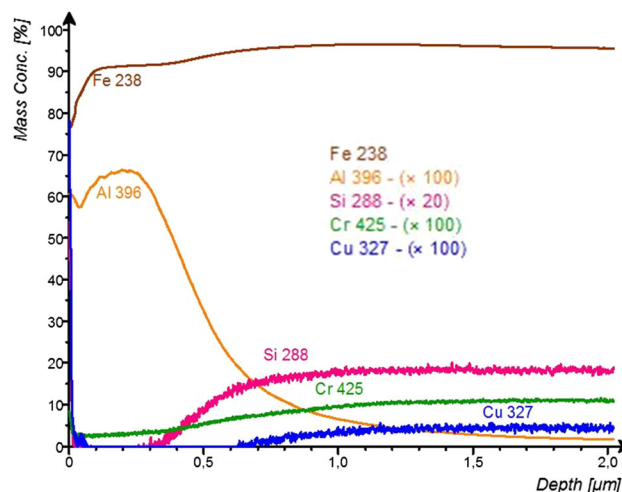
The weight concentration of selected elements with respect to a time profile analysis and the amount of the sputtered material, called “depth analysis”, in which the concentration reaches a constant value, have been used for characterization of the friction layer.



**Fig. 4** GDOES profile analysis of disc A before friction test



**Fig. 5** GDOES profile analysis of disc A after friction test



**Fig. 6** GDOES profile analysis of disc B after friction test

From Table 1, it is apparent that the elements Si, Cu and Al should enrich the friction layer of disc A, while Cr should be decreased. Friction layer of disc B should be depleted of Si, Cr and Cu, while Al should be enriched. (Note: The brake pads wear tested with disc B was determined as higher in comparison to the brake pads wear used with the disc A).

The result of profile analyses of brake discs A and B satisfies the assumptions. Profile analysis of brake disc A (Fig. 5) shows varying concentrations of Si in the friction layer, while this layer is depleted of Cr. Elements Cu and Al show the opposite trend.

Friction layer of the brake disc B (Fig. 6) is deprived of the elements Si, Cr and Cu. In contrast, Al is enriched in the friction layer. Differences in the depth from the surface, when the concentration reaches a constant value, show that Al probably enriches the base material of the brake disc as well.



**Table 2** Chemical composition of the brake discs during friction test

Depth, $\mu\text{m}$	Mass concentration (%)											
	Disc A before friction test				Disc A after friction test				Disc B after friction test			
	Si	Cr	Al	Cu	Si	Cr	Al	Cu	Si	Cr	Al	Cu
0.1	1.20	0.10	0.03	0.07	1.85	0.05	0.19	0.30	0.00	0.02	0.63	0.00
0.2	1.15	0.11	0.02	0.07	1.70	0.07	0.13	0.15	0.00	0.03	0.66	0.00
0.3	1.10	0.12	0.02	0.06	1.50	0.09	0.08	0.10	0.00	0.04	0.63	0.00
0.4	1.05	0.12	0.02	0.06	1.35	0.11	0.05	0.09	0.20	0.05	0.50	0.00
0.5	1.05	0.13	0.01	0.06	1.25	0.12	0.04	0.08	0.45	0.05	0.33	0.00
0.6	1.05	0.13	0.01	0.06	1.20	0.13	0.03	0.07	0.63	0.06	0.21	0.00
0.7	1.05	0.13	0.01	0.06	1.15	0.13	0.02	0.07	0.75	0.07	0.15	0.01
0.8	1.05	0.13	0.01	0.06	1.15	0.14	0.01	0.07	0.85	0.08	0.11	0.03
0.9	1.05	0.14	0.01	0.06	1.15	0.14	0.01	0.07	0.88	0.09	0.09	0.04
1.0	1.05	0.14	0.01	0.06	1.10	0.14	0.01	0.06	0.98	0.10	0.08	0.04
1.2	1.05	0.14	0.01	0.06	1.10	0.14	0.01	0.06	0.98	0.11	0.05	0.05
1.4	1.05	0.14	0.01	0.06	1.05	0.14	0.01	0.06	0.98	0.12	0.03	0.05
1.6	1.05	0.14	0.01	0.06	1.05	0.14	0.01	0.06	0.98	0.12	0.02	0.05
1.8	1.05	0.14	0.01	0.06	1.05	0.14	0.01	0.06	0.98	0.12	0.01	0.05
2.0	1.05	0.14	0.01	0.06	1.05	0.14	0.01	0.06	0.98	0.12	0.01	0.05

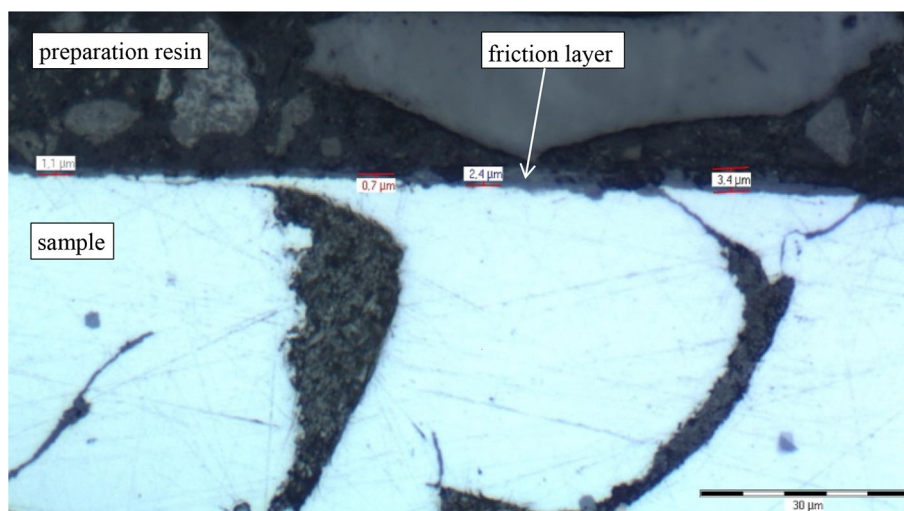
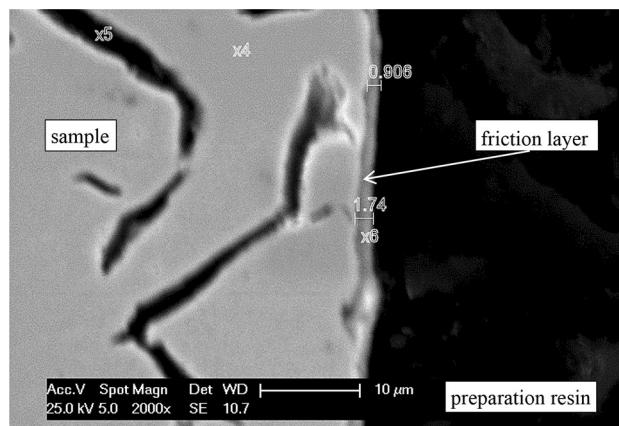
**Fig. 7** Image of brake disc A—cross section, optical microscopy

Table 2 is made of results of GDOES profile analysis depicted in Figs. 4, 5 and 6, the values are subtracted from the graphs (divided by the number in brackets in the graph legend).

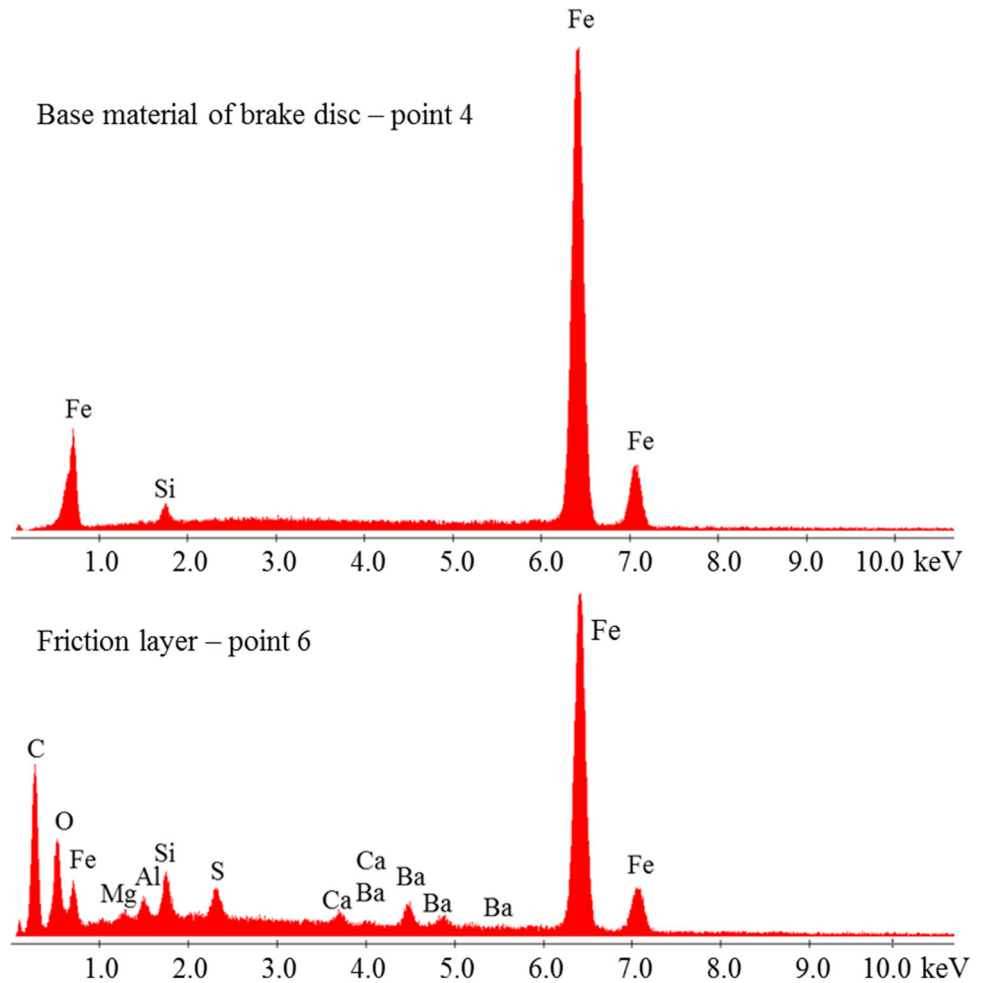
### Characterization of friction layer by SEM with EDX analysis

Cross-sectional samples of brake discs were prepared for observation of friction layer, followed by EDX analysis and estimation of the thickness of the friction layer by SEM and by optical microscopy.

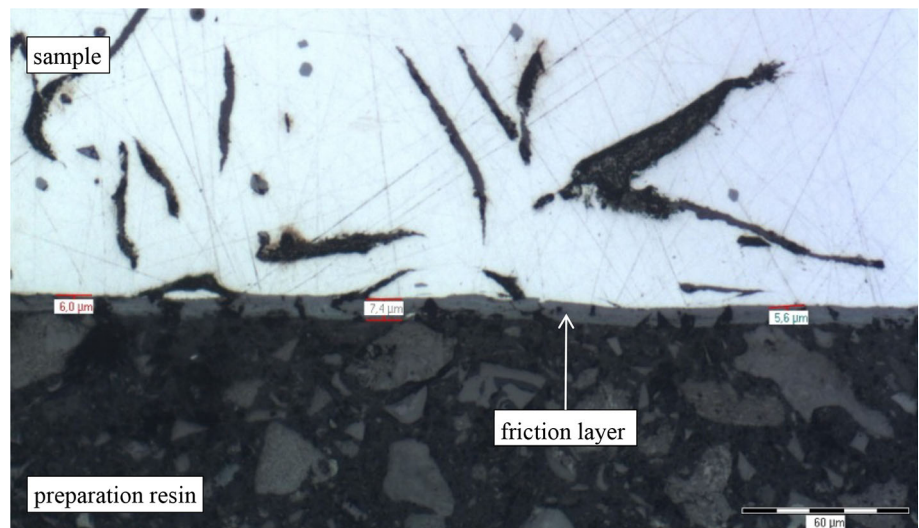
Friction surface layer of the brake disc manifests a different morphology compared to the base material and a

**Fig. 8** SEM image of brake disc A

**Fig. 9** EDX analysis of base material and friction layer of brake disc A

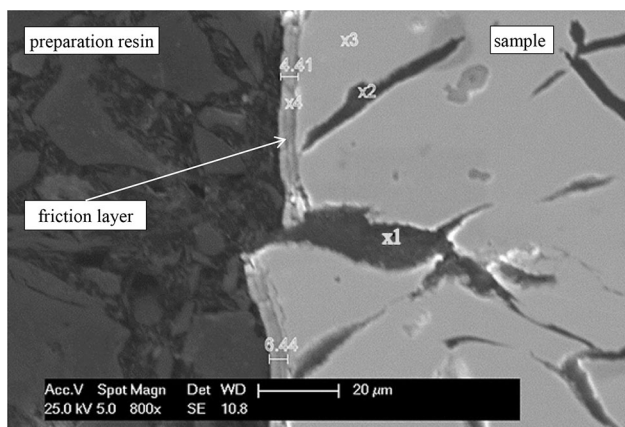


**Fig. 10** Image of brake disc A—optical microscopy



different chemical composition as well. As already discussed, in terms of the chemical composition, the friction layer deposited on the brake disc should be enriched by selected element (-s), coming from the brake pads during friction process.

Figure 7 shows the friction layer with variable thickness on the surface of the brake disc A, observed by optical microscopy. The presence of friction layer is also visible in the SEM image (Fig. 8) and it is confirmed by EDX

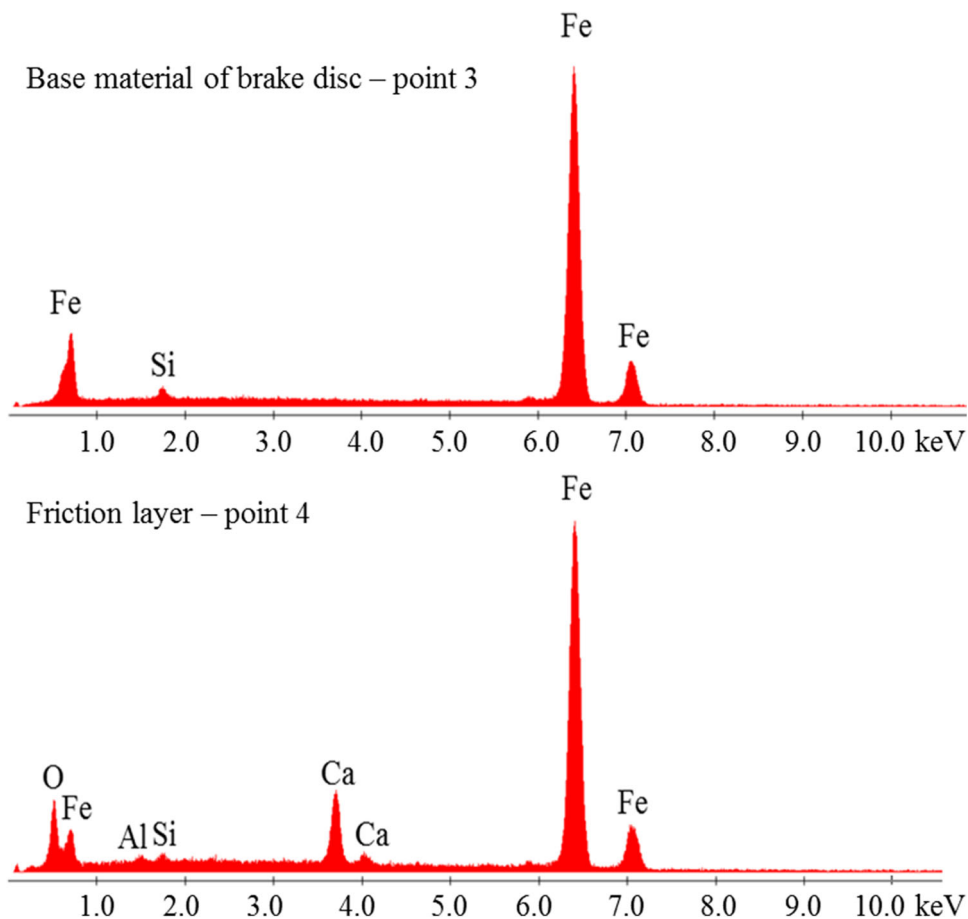


**Fig. 11** SEM image of brake disc B

analysis (Fig. 9). The places where the EDX analysis was carried out are indicated in Fig. 8.

In the base material of the brake disc, Si (selected as a marker for friction layer) and Fe which is base element of cast iron (see Fig. 9) were detected. Unlike the base material, the friction layer is enriched with Al and Si (the markers) and also with S, C, O, Mg, Ca and Ba (the other elements).

**Fig. 12** EDX analysis of base material and friction layer of brake disc B



The thickness of the friction layer is ranging from 0.7 to 3.4 microns as estimated by the evaluation of the images obtained using SEM and optical microscopy. There are also places without friction layer on the surface of the disc.

In comparison to friction layer deposited on the disc A, the friction layer on disc B is visible even at lower magnification (Figs. 10, 11) and its chemical composition was confirmed by EDX analysis (Fig. 12) made on the surface of the disc.

EDX spectra of the brake disc B base material (Fig. 12) Si (marker of the friction layer) and Fe and Mn were detected. Friction layer originated at the friction surface of disc B after the friction test is enriched with Al (marker element) in comparison of the base material (see Fig. 12); moreover, Ca and O were also detected by EDX. Silicon is presented in the base material (matrix) of the disc and in the friction layer as well, presenting no change.

Based on performed analysis by SEM and optical microscopy, the thickness of the friction layer formed on the disc B is ranging from 4.4 to 7.4 microns. There are also places without this layer on the surface of the disc.

The following table (Table 3) summarizes the thicknesses of friction layers of both discs determined from all

**Table 3** The thickness of the friction layer

Method	Friction layer ( $\mu\text{m}$ )	
	Disc A	Disc B
GDOES	0.5–0.7	1.4–1.9
SEM, OM	0.7–3.4	1.7–7.4

the measured spectra and microstructure images (including those not listed in this article).

## Conclusions

The aim of the paper is to evaluate the suitability of GDOES for characterization of friction layer formed on the surface of brake disc. It was preceded, however, by the definition of conditions for the chemical composition of the friction layer, based on the knowledge of the chemical composition of pairs (brake pads and brake disc) and on the friction layer formation mechanism. If the content of the chemical elements in the brake pad is higher than in the disc, these elements should enrich the friction layer of the brake disc, and vice versa. Together with this assumption, it has been defined that it is required to use selected chemical elements as indicators (markers) of the friction layer and also to estimate the thickness of the friction layer. Si, Cr, Cu and Al were selected as indicators of friction layer, and also as suitable elements for the estimation of thicknesses of the friction layer. Based on these assumptions, the thickness was estimated and the composition of friction layers of both discs using GDOES spectrometry was determined.

Furthermore, the GDOES results were compared with microscopy measurement. It was observed that the thickness of the friction layer originated on the surface of brake disc and the thickness evaluated by GDOES profile analysis and by both microscopy techniques (optical microscopy and SEM-EDX) differ from each other. While in profile analyses, the results were generally in hundreds of nanometres, in the case of scanning electron and optical microscopy, the results ranged in units of micrometres. The differences were probably caused by the formation of uneven friction layer. Areas without friction layer have also been observed. This could be caused by the occurrence of graphite with its lubricating properties, which decreases the adhesion of wear particles to the surface of the brake discs.

GDOES analyzes a circular area with a diameter of 4 mm; thereby this method minimizes the influence of the occurrence of sites with less or no friction layer.

Based on the results, it can be said that the GDOES is a suitable method for the characterization of friction layers.

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