Characterization of Friction Layer of Brake Pad Using Scanning Electron Microscopy and Raman Microspectroscopy

Charakterizace frikční vrstvy brzdové destičky pomocí skenovací elektronové mikrokospie a Ramanovy mikrospektroskopie

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Wear of friction materials symbolizes a challenge since several chemical compounds are being released during the braking process. Usually, it is very difficult to identify and describe the source of pollutant originated by vehicle operation. It can be said that wear debris shall be present in the friction layer of brake pad adhering to the rotor. Due to the fact that there is still a lack of information about chemical composition of initial brake pad, composition of friction layer and the wear debris composition, it is necessary to develop analytical methods, which make it possible to correlate microstructure and chemistry of the friction layer. In this study scanning electron microscopy, energy dispersive X-ray microanalysis with Raman microspectroscopy were introduced as a possible combination of analytical techniques. It provided information about morphology, elemental and phase composition of the friction layer.

***Key words**: brake pads; friction layer; electron microscopy; Raman microspectroscopy*

Studium opotřebení frikčních materiálů představuje naléhavou problematiku s ohledem na skutečnost, že se během tohoto procesu se do okolí uvolňuje řada chemických sloučenin. Často je velmi obtížné identifikovat a popsat zdroj tohoto znečištění pocházejícího z provozu automobilu. Otěrové částice by měly být přítomny ve frikční vrstvě brzdové desky, která přiléhá k rotoru. S ohledem na skutečnost, že pořád není dostatek informací o vztahu mezi chemickým složením vstupní brzdové desky, složením frikční vrstvy a složením otěrových částic, je třeba vyvinout analytické metody, které umožní korelovat mikrostrukturu s chemickým složením frikční vrstvy. Tento příspěvek představuje spojení skenovací elektronové mikroskopie, energiově disperzní RTG mikrospektroskopie a Ramanovy mikrospektroskopie jako možnou kombinaci analytických metod. Jejich spojení poskytuje informace o morfologii, prvkovém i fázovém složení frikční vrstvy.

***Klíčová slova**: brzdové destičky; frikční vrstva; elektronová mikroskopie; Ramanova mikrospektroskopie*

The brake pad is a multicomponent system, which is typically created by more than 10 constituents – reinforcing agents, abrasives, lubricants, binders and fillers [1]. Phenolic resin is used as a matrix and several metals, ceramics, minerals, carbons or polymers are present in the typical brake pad. Exact formulation is know-how of producer and it variates according to applications. Several studies presented chemical composition and study of the morphology of wear particles released from brake pads [2, 3]. It was described that chemical composition of wear debris is almost similar to the chemical composition of friction layer. Distribution of constituents in friction layer is crucial for the braking process. High pressure and temperature occur during braking, therefore various changes of morphology and chemistry can occur. Formation of friction layer in respect to wear performance has been studied [4] and it revealed that particles, which escaped the friction interface create the friction layer. Electron microscopy made it possible to uncover [2, 5, 6] that the presence of smaller particles has a high influence on wear debris emission. Authors Malachová et al. [7] studied toxicity and mutagenicity of wear debris and found that wear debris were acutely toxic but mutagenicity was not observed.

Properties of the friction layer determine the friction performance of the brake pad. However, there is still lack information about the relationship between the chemical composition of initial pad formula and composition of friction layer, and also between the composition of friction layer and wear debris composition. Additional studies of friction layer formation and its contribution to brake process are therefore necessary. The aim of this study is to propose new possible combination of analytical methods focused on mapping of friction layer, which would enable the correlation of microstructure and the chemistry of the friction layer.

Materials and methods

Model brake pad

A model pad of the following composition (CuS, CuZn, CuSnP, Sn, iron fibers, graphite, coke, aramid fibers, $BaSO_4$, ZrO_2, $FeCr_2O_4$, SiO_2, SiC, MgO, ZnO, MoS_2, Al_2O_3, SnS, rubber, phenolic resin) was prepared and tested according to the procedure ISO 26867:2009 Road vehicles -- Brake lining friction materials -- Friction behavior assessment for automotive brake systems was used for the automotive brake dynamometer test [8]. Brake dynamometer tests were performed using the Compact Brake Dynamometer Link M2800 in an environmental chamber with wind tunnel simulating corresponding air flow. The original equipment Škoda Octavia I cast-iron disc (280×22) and caliper (FS-III) were used in dynamometer test.

Microscopic analysis

Surface/friction layer was analyzed by use of scanning electron microscope (SEM) Quanta FEG 450 (FEI) equipped with EDS analyzer Apollo X (EDAX).

Raman spectra of surface/friction layer were obtained using Smart Raman Microscopy System XploRA ™ (HORIBA Jobin Yvon, France) with integrated light microscope Olympus BX41/51. Raman spectra were acquired with 532 nm excitation laser source, and 1 200 g·mm^{-1} grating.

Results and discussion

Scanning electron microscopy is a useful tool for study and characterization of friction layer of brake pads. Typical friction layer is displayed in Fig. 1.

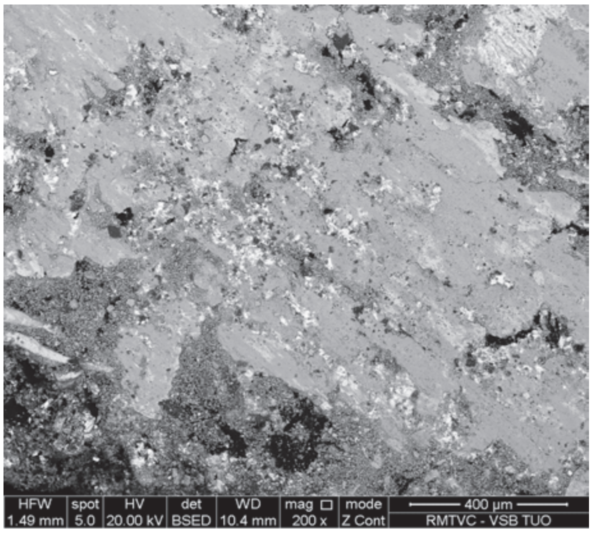

Fig. 1 Image from SEM of friction layer of the tested brake pad
Obr. 1 Obrázek ze skenovacího elektronového mikroskopu zobrazující frikční vrstvu testované brzdové desky

Back scattered electron imaging enables us to see material contrast in the studied sample. Metals, such as iron, are shown as bright white where carbon-based compounds are black. Shades of black-gray-white depend on the relative atomic mass of components. Without further EDX analysis, it is almost impossible to distinguish each components of the pad. Therefore EDX mapping was performed (Fig. 2).

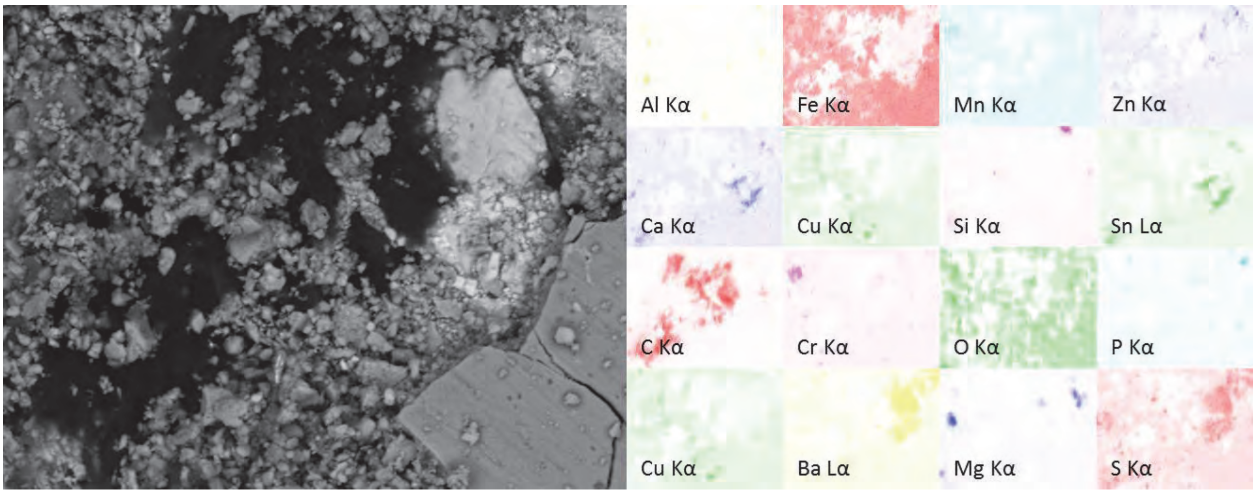

Fig. 2 Image from SEM (left) and EDS maps of selected area (right) of pad after breaking procedure (magnified selected area from figure 1)
Obr. 2 Obrázek pořízený skenovacím elektronovým mikroskopem (vlevo) a EDS mapy (vpravo) vybrané části brzdové desky po brzdném procesu (zvětšená oblast z obrázku 1)

From a comparison of EDX maps of elements, it is possible to deduce the chemical origin of particles or aggregates. For instance, the map of barium and the map of sulfur correlates together and therefore it can be said that the particle is created by barite. However, it is not always possible to determine all particles due to the overlapping of maps and signal in general. Therefore additional analysis, which enables determination of phases, is needed.

Raman microspectroscopy allows phase point analysis and it revealed (Fig. 3) the presence of iron oxides (mainly Fe_2O_3, and Fe_3O_4), barite ($BaSO_4$), graphite, amorphous carbon, molybdenite (MoS_2), aluminum oxide (Al_2O_3), copper oxide (Cu_2O) in the friction layer of the tested brake pad. Apart from point analysis also analysis of selected area can be studied using Raman micro-spectroscope. That kind of analysis is called Raman mapping.

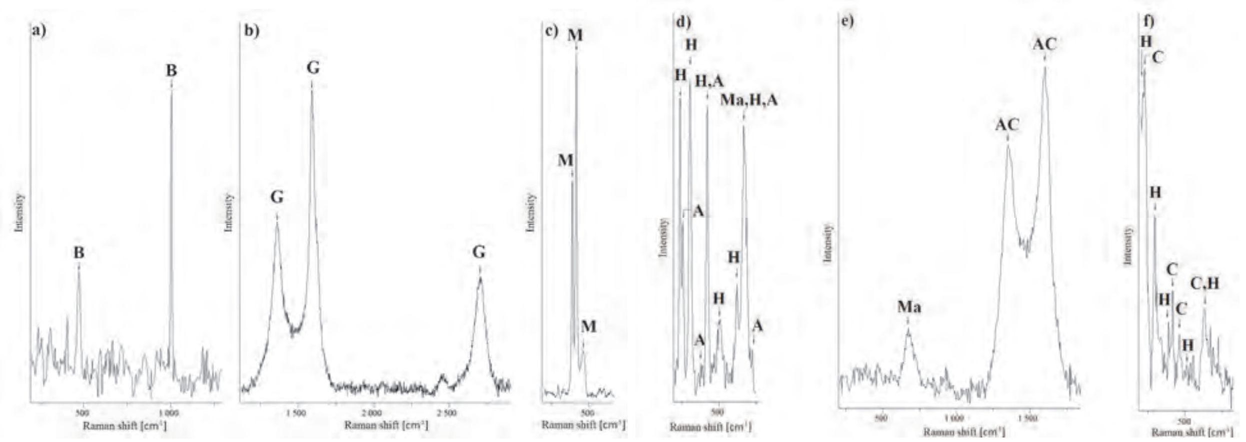

Fig. 3 Raman spectra of brake pads after braking process a) barite (B), b) graphite (G), c) molybdenite [MoS_2] (M), d) hematite [Fe_2O_3] (H), aluminum oxide [Al_2O_3] (A), and magnetite [Fe_3O_4] (Ma), e) amorphous carbon (AC), and magnetite [Fe_3O_4], f) copper oxide [Cu_2O] (C), and hematite [Fe_2O_3] (H)

Obr. 3 Ramanova spektra brzdové desky po brzdném procesu a) baryt (B), b) grafit (G), c) molybdenit MoS_2 (M), d) hematit Fe_2O_3 (H), oxid hlinitý Al_2O_3 (A) a magnetit Fe_3O_4 (Ma), e) amorfní uhlík (AC) a magnetit Fe_3O_4, f) oxid měďnatý Cu_2O (C) a hematite Fe_2O_3 (H)

Fig. 4a displays friction layer of brake pad under a light microscope, where selected area is marked. Spectral map of the selected area is in Fig. 4b, where each detected compound in real size and distribution in the friction layer can be seen. Red color represents graphite, green color represents silicon carbide and blue color represents zirconium oxide. The rest of the friction layer in the selected area was created by amorphous carbon, which is represented by black color. According to the comparison with the scale bar, it can be assumed that graphite particles are approximately 1µm wide. Some particles consisted of several compounds. For example silicon carbide (green) is surrounded by graphite (red), as well as zirconium oxide (blue) contained a small amount of graphite. The selected analyzed area was created mainly by amorphous carbon (black).

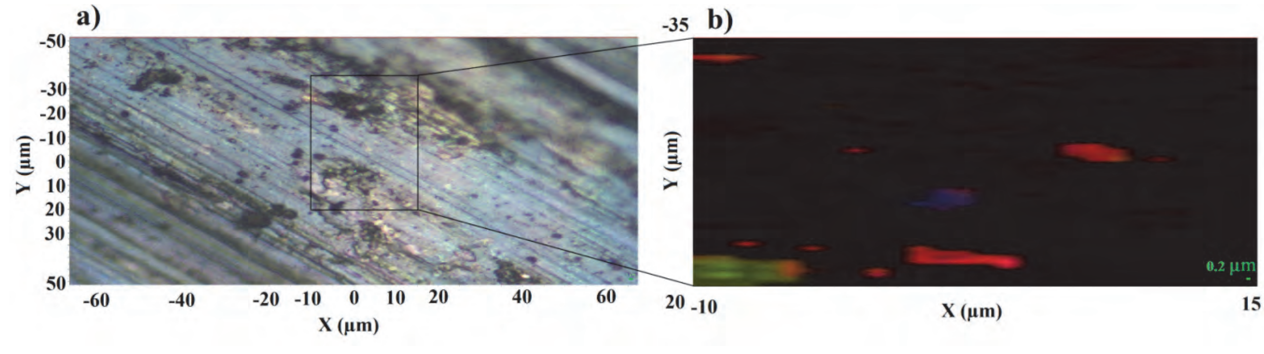

Fig. 4 Light microscopy image of the brake pad sample with marked selected area (a), corresponding spectral map (b): red color represents graphite compounds, green color represents silicon carbide, and blue color represents zirconium oxide. The black color is mainly amorphous carbon.

Obr. 4 Obrázek ze světelného mikroskopu brzdové desky s označením vybrané oblasti (a) s odpovídající spektrální mapou (b): červená barva reprezentuje složky grafitu, zelená barva reprezentuje karbid křemíku a modrá barva reprezentuje oxid zirkoničitý. Černá barva je vesměs amorfní uhlík.

Conclusions

This pilot study showed that the combination of scanning electron microscopy, EDX, and Raman microspectroscopy is useful for friction layer characterization. The combination of scanning electron microscopy, EDX, and Raman microspectroscopy is useful for friction layer characterization. The combination of these methods enables studying of the sample morphology, elemental and phase composition. A better understanding of the process of friction layer formation and distribution of components in friction layer is important in terms of wear debris emission and its reduction.

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ArcelorMittal vidí ocel jako „perfektní materiál pro hospodářství s kruhovým oběhem“

Stahl Aktuell *22.11.2016*

Až o 3 gigatuny za rok se dají snížit emise CO_2, když by byl skleníkový plyn v meziodvětvové míře zachycován a v průmyslovém měřítku dále využíván. To je jeden z prvních výsledků studie, kterou podporuje a zadává ArcelorMittal a řada dalších výrobních a poradenských firem k tématu lepšího využití průmyslových odpadních plynů v ocelářském a cementárenském odvětví, jakož i v chemickém průmyslu. Výsledky studie byly představeny na světové klimatické konferenci v Marrakeši v Maroku.

Evropa: pochybnosti o prosaditelnosti zvýšení cen oceli

Stahl Aktuell *24.11.2016*

V uplynulých týdnech ohlásili výrobci oceli z EU částečně i velmi výrazné zvýšení cen za své výrobky. Jako důvod jsou uvedeny většinou stoupající náklady na suroviny. Analytici švýcarské banky UBS ale pochybují o tom, zda se tyto požadavky dají v plné výši realizovat. „Tyto požadavky se objevují v tradičně slabém ročním období“, píše bankovní dům. Koncem roku odbourávají totiž mnozí odběratelé oceli své skladové zásoby. Například ArcelorMittal požaduje pro prosincové dodávky za široký pás, válcovaný za tepla cenu 550 € na tunu. Zákazníci už ovšem tuto cenu stlačili na úroveň 500 € za tunu.

Největší filtrační zařízení na světě

Rheinische Post *29.11.2016*

Na továrním pozemku Thyssen v Duisburgu-Schwelgern se momentálně staví s pomocí speciálního jeřábu 100 m vysoký průmyslový komín. Je součástí nového odprašovacího zařízení, které bude dáno do provozu na jaře 2017 a bude celosvětově největším odprašovacím zařízením pro sintrovací jednotku. Zařízení s plachetkovým filtrem má dále zlepšit životní prostředí v Duisburgu.