

## Tribology and surface characterization of brake materials

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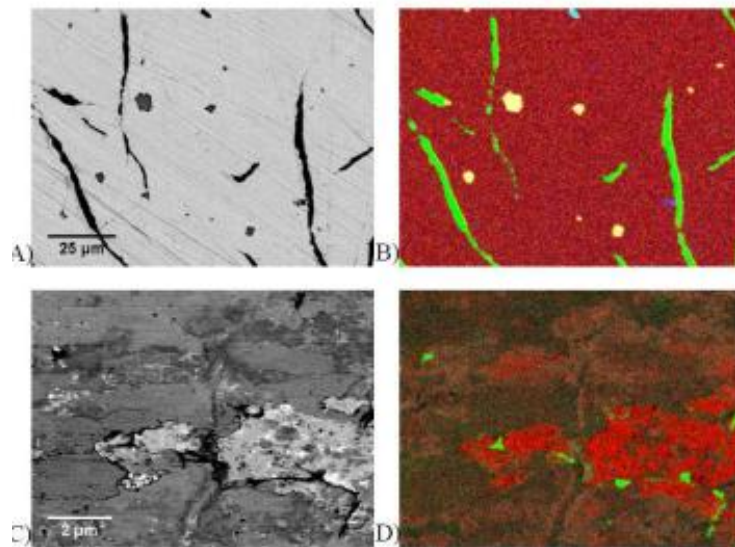
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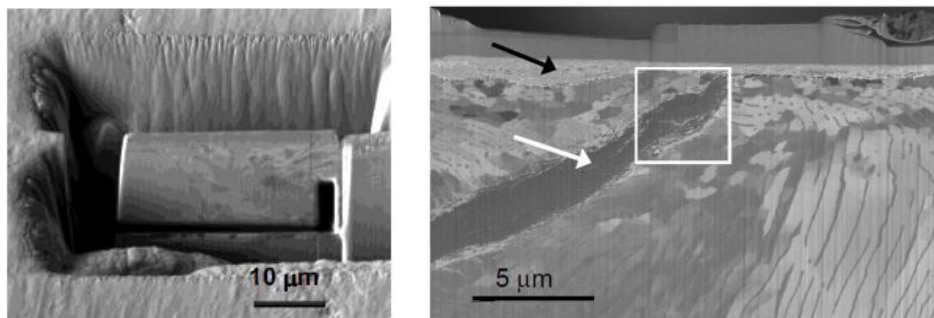
The lining materials of brakes pads are composites formed by hot compaction of powders including different raw materials (more than 20). These components include i) binder, that holds all components together and forms a thermally stable matrix; ii) structural materials, providing mechanical strength. Materials used are metal, carbon, glass, and/or kevlar fibres and more rarely different mineral and ceramic fibres; iii) fillers, to reduce cost and improve manufacturability. Different minerals such as mica and vermiculit are employed. Barium sulphate is another used filler; iv) frictional additives, to ensure stable frictional properties and to control wear rates of both pad and disc. The material of the brake discs is grey cast iron with 3–4 wt.% carbon. This material contains small flakes of graphite in a pearlitic matrix. Tests were performed using an automotive brake full scale dynamometer tests using AK-Master Procedure.

The friction film is observed at the surface of the cast iron disc after dynamotor tests as shown in Figure 1.



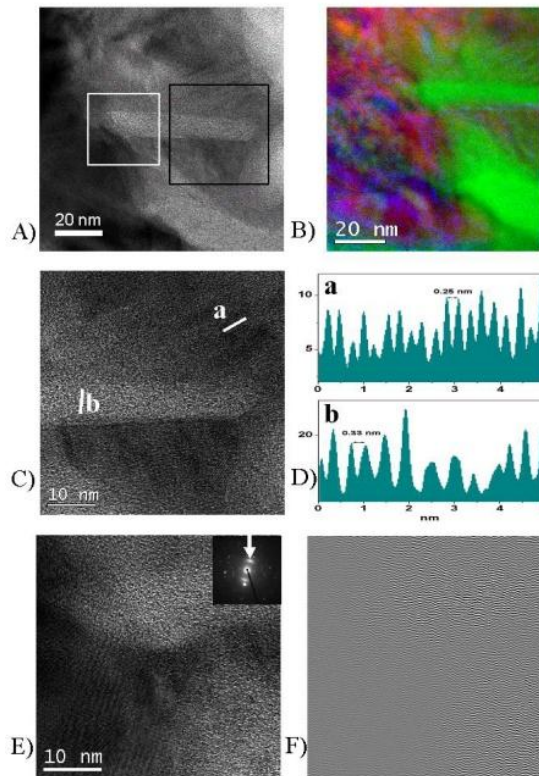
**Figure 1.:** Disc surface (A) BEI of the pristine grey cast iron disc revealing graphite flakes (black) in a ferritic/pearlitic matrix (light grey); dark grains are MnS<sub>2</sub>. (B) Composite EDS-map (iron: red, graphite: green, Mn+S: light yellow, silicon: blue, aluminum: cyan). (C) BEI of disc surface after the AK-Master friction test. Dark grey surface film (third body) is composed mainly of magnetite and graphite. Light grey areas are exposed iron; bright spots correspond to tin sulfide or barium sulfate from the PMC pad. (D) Composite x-ray map (graphite: green, iron: red, oxygen: cyan, Fe + O: salmon).

In order to observe this film with Transmission Electron Microscopy (TEM), samples were prepared by using Focused Ion Beam (FIB) system as shown in Figure 2.



**Figure 2.** Ion-induced secondary electron image of the TEM lamella still bound to the disc surface. Ion-induced secondary electron image of the TEM lamella. The black arrow indicates the friction film beneath the platinum protective strip. The white arrow indicates the graphite flake diagonally crossing the plastically deformed matrix of the cast iron. The white square indicates the main region of TEM analysis.

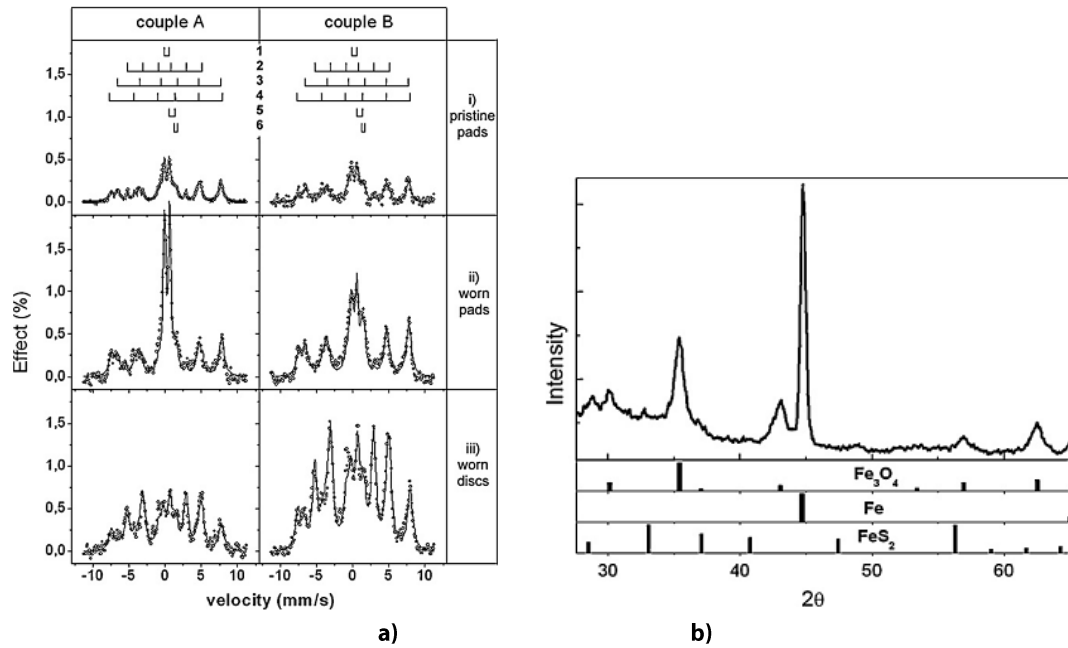
The results of TEM presented in Figure 3 show graphite flakes of a grey cast iron brake discs became heavily disordered, with wrinkled few-layer graphenes wedged between nanoparticles of iron and magnetite, due to nanoscale mechanical cutting that occurred during the macroscopic shearing process of braking tests. FIB and EFTEM captured the process of graphite exfoliation and magnetite formation, revealing that the oxidation was fostered by the cracking due to differences in elastic moduli of iron and graphite and by the dissecting of iron into nanoparticles by graphene cutting edges. The latter mechanism will be supported further by oxidation. The anisotropic shear modulus in the graphite crystalline structure caused the delaminating of few-layer graphenes that penetrated the iron bulk, peeling off iron nanoparticles. On the other hand, the low adhesion between graphite basal planes allowed the exfoliation of few-layer graphene batches that were wedged apart by minute iron or magnetite particles [1].



**Figure 3. Micrographs, profiles, and maps of graphene exfoliation. (A) EFTEM image of a graphite rod and exfoliating graphene batches between iron-bearing grains. Black square indicates area of Figure 5 C, white one indicates Figure 5 E. (B) EFTEM composite element map (iron: red, carbon: green, oxygen: cyan). (C) High resolution micrograph of graphite between magnetite grains. Lines labeled a and b indicate the location of the contrast profiles at the right. (D) Profile a is compatible with the interplanar distances of magnetite, profile b is few-layer graphene. (E) High resolution EFTEM micrograph of the white square indicated in Figure 5 A, SAED pattern inset. (F) Inverse FFT of the graphite spots (white arrow on the SAED), showing wrinkled lattice planes.**

Mössbauer spectroscopy proved to be extremely useful in the identification of iron-bearing phases, the shallow depth and large area sampling of CEMS permitted representative analysis of films on tribo-surfaces. The combination of XRD with Mössbauer Spectroscopy (see Figure 4) identified all iron-bearing compounds on the surface of complex friction couples, like PMC pad and cast iron disc counter parts, even those with low crystallinity [2].

Several publications have emphasized the role of friction films, however no reference was found concerning the presence of pyrite as part of the third body. The origin of iron sulfides has to be more thoroughly investigated and the sulfur source identified. The observation of pyrite in the film opens new questions about its effect on the tribological behavior of brakes and its presence has to be considered in amore realistic description of the braking condition. Pyrite, magnetite, and wüstite were positively identified I all friction films, and hematite was ruled out as a major phase. It was shown that iron copper sulfides and iron oxide–hydroxides decomposed under frictional load.



**Figure 4. a) CEMS spectra from (i) pristine pads (ii) worn pads and (iii) worn discs. MS peak positions indicated on top refer to (1) pyrite, (2) α-iron, (3 and 4) magnetite, and (5 and 6) wüstite. Same %effect scale on all spectra. b) GIXRD (incidence angle 1°) from the disc surface after the dynamometer test shows the presence of nano-crystalline magnetite (Fe<sub>3</sub>O<sub>4</sub>), α-iron (Fe) and minor amounts of pyrite (FeS<sub>2</sub>).**

Based on the experimental observations of the group, computer simulation are under developmet by the Computer Simulation Group from Physics Institute at UFRGS composed by Dr. Leonardo Brunnet, Dr. Sebastian Gonçalves and Dr. Evy Salcedo (UFSC).

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