MICROSTRUCTURAL CHARACTERIZATION OF LOADED BRAKE MATERIALS

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SUMMARY
A conventional organic-base brake lining was rubbed against different metallic counter bodies in order to study stick-slip phenomena and correlate them with the formation of so-called friction layers. A special test rig was designed for this purpose and analytical electron microscopy was applied to reveal microstructural and chemical changes at the surface, in superficial zones and debris particles. Tribooxidation at the metallic surface was identified and the transfer of metal oxides to the pad was evidenced. Though metal oxides as the major wear particle fraction determined the phase structure of the friction layer, compositional mixing with the mineral components of the pad was observed as well. The dominating role of the metal oxides led to different evolutions of static and dynamic coefficients of friction during the tests with one pad material against different counter bodies.

Keywords: brake, stick-slip, friction layer, tribooxidation, TEM

1 INTRODUCTION
During the operation of a frictional brake, kinetic energy is transformed to other forms of energy, mainly heat, which induces structural and chemical changes at the surfaces of both counterparts of the tribological system. Brake experts are convinced that good performance of a brake depends on the formation of a so-called friction layer at the pad surface. Only very little is known up to now about the structure of such friction layers, and how the properties are influenced by the composition of the counterpart materials and by stressing conditions.

Some authors observed a transfer of iron from the disk to the pad surface [1]. In order to show this more clearly, we used a friction material without steel chips or iron oxide particles and pressed it against a rotating steel disk in one of our previous studies [2-5]. In addition to iron transfer, compositional mixing of all components of the tribological system took place. Such layers seem to be typical if the situation is like this that a high quantity of filler particles is released from the pad by wear [5]. If that is not the case, iron oxide from the disk seems to be the main material, forming patches of friction layer around reinforcing elements like glass or steel fibres [6].

In our previous studies [2-5], the type of material forming the friction layer was the same, irrespective of stressing conditions, provided that the same pad and disk materials were used. In this work, we intended to investigate the influence of specific disk materials on friction layers produced with the same pad. A very low sliding velocity was applied in a test rig which was designed especially for studying stick-slip situations. Since brake groan can be attributed to this phenomenon [7], such studies are of interest not only for academic, but also for industrial means.

2 EXPERIMENTAL PROCEDURE
The pad material used in this study was a polymer matrix composite (PMC), provided by Honeywell-Jurid, Hamburg. Since it is an industrial material, the exact formulation cannot be given here. As in most friction materials of this type, 6 classes of components can be distinguished. The major component of each class was: 1. glass fibres as reinforcing element, 2. chromium oxide as abrasive, 3. calcite as filler, 4. several metal sulfides as friction modifiers, 5. phenolic resin as binder and 6. aramid pulp as fibrous component.

Six metallic counter-bodies were regarded, namely: steel St52-3, Cr-coated St52-3, aluminium, steel casting GS60 and cast iron types GG15 and GG30. Here we will concentrate mainly on results obtained with steel and aluminium counter-bodies.

Figure 1 shows the experimental set-up for distinct measurements of the stick-slip transition.

Figure 1: Schematic representation for measurements of the stick-slip transition
A piece of lining material is pressed with a constant contact force against the metallic counterpart, which is constructed like a slide. If the tangential force exceeds a
certain critical value, the slide begins to move and the coefficient of friction will drop from its high static value to a lower kinetic one. The difference of both is a measure for the propensity of the system to frictional vibrations i.e. noise production. This process can be repeated many times, and the changes, induced by the formation of patches of the friction layer will be recorded. Changes of $\mu_s$, $\mu_k$ and $\Delta\mu = \mu_s - \mu_k$ can be evaluated and attributed to changes of the surface structure.

Several microscopic and spectroscopic techniques were applied for characterizing the surface, the superficial zones and debris particles. A scanning electron microscope (SEM) equipped with an energy-dispersive X-ray spectrometer (EDX) was used to reveal surface features. Cross-sectional transmission electron microscopy (XTEM) combined with selected area electron diffraction (SAD) and EDX was applied to examine the friction layer on the pad in great detail. Thin cross-sections were prepared by face to face gluing, tripod polishing and ion beam thinning. Debris particles were scratched off the worn surface, suspended on carbon-coated support grids and investigated directly in the analytical TEM (JEOL 4000FX).

3 RESULTS AND DISCUSSION

The evolution of the static and dynamic coefficient of friction with the number of cycles is shown in Figure 2 for sliding against steel, chromium and aluminium.

![Figure 2](image)

**Figure 2:** Static and dynamic coefficient of friction for sliding against a) St52-3, b) Cr, c) Al

Though the stationary values of $\mu_s$ seem to stabilize somewhere between 0.5 and 0.6 for all counter-bodies, the evolution before reaching this point is quite different. For the iron-base materials – they all behave similar, thus only one is shown - $\mu_s$ stabilizes already after 500 cycles. For chromium and aluminium there is a continuous increase so that we cannot be sure that the steady state has been reached after 5000 cycles. The highest value of $\Delta\mu$ appears with steel and the lowest one with aluminium.

Macroscopic views of the steel and aluminium surface are shown in Figure 3.

![Figure 3](image)

**Figure 3:** Macroscopic views of two counter bodies

Dark patches on the counter body surfaces suggest that tribooxidation had taken place. This was confirmed by EDX-analysis in the SEM at sites marked by arrows in Figure 3. The micro-topography of pad, steel and aluminium counter body, as revealed in the SEM, is shown in Figure 4.
Figure 4: SEM-micrographs of a) pad rubbed against steel, b) from steel counter body and c) from aluminium counter body

The most characteristic feature of the worn pad surface are patches of compacted debris, about 10 – 20 µm in diameter. The surface of such patches is smooth, whereas the surrounding areas, comprising mainly loose particles, are rough. The metallic surfaces generally are more uniform with some occasional scratches from abrasive particles. The bright features are loose debris particles comprising a compositional mix of pad components and metal oxides. In the case of aluminium (alloy) the debris contains high amounts of Zn and Mg. Figure 5 shows a cross-sectional view of a friction layer adhering to a steel chip of the pad (identified clearly by EDX) after rubbing against the steel counter body.

Figure 5: Cross-sectional TEM-micrograph from friction layer (top) and substrate (bottom) after rubbing against steel counter body and SAD-pattern from friction layer

The corresponding SAD-pattern is shown in Figure 5. It indicates a nanocrystalline structure. Parts of the layer could be identified as α-Fe. The inner ring diameters correspond to prominent lattice planes (110), (200), (211) and (220) of α-Fe. The corresponding EDX-spectrum is shown in Figure 6. It clearly reveals a compositional mix of all components of the tribological system, oxide particles of mixed composition were identified by debris investigation in the TEM. One such particle is shown in Figure 7. Debris particles formed with the aluminium counter body could be attributed to aluminium oxide or aluminium-zinc oxide phases. An example is shown in Figure 8. The SAD-pattern again reveals the nanocrystalline microstructure (Figure 8).
In accordance with our previous studies [2-5] we can formulate the following hypothesis: friction layer are formed by mechanical alloying of wear particles, while the crystallographic phase of the major wear fraction is preserved.

4 CONCLUSIONS

Static as well as dynamic coefficients of friction depend on the formation of patches of a friction layer on the pad surface. Due to the growth of contact areas, the coefficients of friction increase gradually during a run-in period.

Since the patches of friction layer are formed by debris compaction, their crystallographic structure and composition will be determined by the wear mechanisms. The two case studies shown here suggest that triboxidation at the surface of the counter body was the controlling process. This can also explain qualitatively the different behaviour of the two tribological systems during the run-in period.

In respect to stick-slip effects aluminium seems to be more favourable than iron-base materials. Unfortunately wear rates of aluminium are much higher (not shown here).

Of course we must admit that forces, velocities and surface temperatures of our tests cannot be compared with values relevant for a real brake application. Therefore we are planning to produce friction layers by braking simulation tests, which then can be analysed in respect to stick-slip behaviour in our test rig.

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6 REFERENCES

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