



## CHANGES WITHIN THE SUPERFICIAL LAYER OF SOME PTFE + GLASS FIBBER COMPOSITES

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### **Summary**

*This paper presents the results concerning changes within the superficial layer, pointed out by the help of X-ray diffractometry. Tests were carried out on roller-shoe tribomodel, with water lubrication and dry regime. The initial and after sliding results are compared. These pointed out the fibber orientation and accumulation within the superficial layer.*

**Keywords:** PTFE + glass fibber composite, water lubrication, X-ray analysis, EDS analysis

### **1. MATERIALS AND TESTING METHODOLOGY**

PTFE composites are recommended for bearing application as they have good tribological behaviour even with poor lubricants as water [3].

There were tested composites with glass fibber into a PTFE matrix. Some of their mechanical properties are given in Figure 1. Materials are available from CEPROINV Focșani, Romania One may notice that Shore hardness is in a narrow range for all tested materials and that traction limit is not a linear function with glass fibber concentration.

This analysis is done for dry regime with  $v=0.5...1$  m/s and a normally applied load of 0.5...1 kN. The water lubrication regime has the same parameters in the range of  $v=1...3$  m/s for sliding speed and of 1...4 kN for the load. The tribomodel was type roller-shoe. The rollers were made of stainless steel of ~40 HRC and they were fine grinded and the shoe has a centre angle of

120°. The testing machine [8] has the following characteristics: rotation speed 100... 2000 rot/min, loading from 1 to 10 kN, monitoring friction moment, load and angular speed, a dynamic acquisition system (Hottinger-Baldwin DMCplus). Tested shoes after sliding 10500 m in water in open circuit at 2.5 m/s are given in figure 2. First-up shoes were bearing 1kN ( $p=0.76$  MPa), the following supporting 2, 3 and 4 kN respectively.

EDS analysis begins with peak identification by the help of spectrometer soft (KLM). The reference sample was pure aluminium. The shoes were cut with low speed cutting regime in order to have no influence on the analysed zone. The middle piece, including the contact zone was selected for analysis. The samples were cleaned with white spirit and then dry. The parameter selected for the spectrometry were time  $t=100$  s, accelerating voltage of the electrons in the spectrometer column was  $U=25$  kV, magnification  $M=25$ . The surface exposed to the EDS analysis was 3.5 x 3.5 (mm x mm).

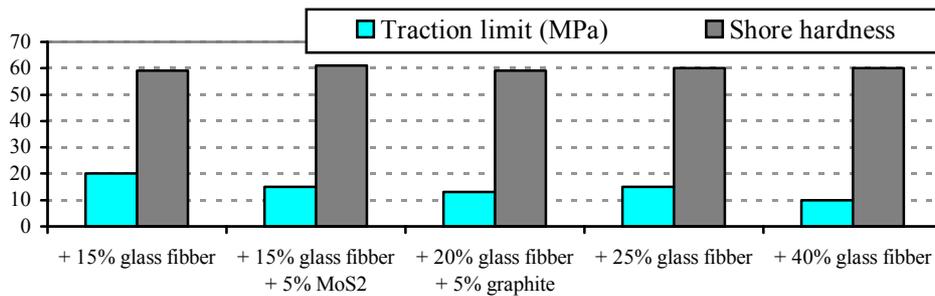


Figure 1

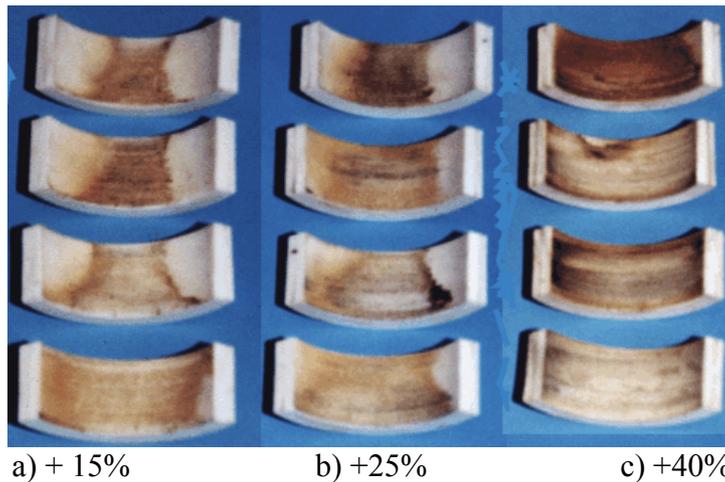


Figure 2 Composites with PTFE matrix (percentage is for glass fiber mass in the composite)

Figures 4 and 5 show the obtained spectrograms for: a) composite sample before testing and b) after sliding in water for 4 steps of loading (each one of 1kN), the final one being of 3kN (an average normal pressure of  $p=3\text{MPa}$  and the sliding speed of  $v=2.5\text{m/s}$ ; the sliding distance was 1500m (~10 minutes) for each loading step. The ordinate of the spectrograms represents the number recorded by the X-ray detector.

For the composite PTFE + 15% glass fiber that after sliding the content of Si is increased, sustaining the idea of fiber accumulation in the tribo-layer. Traces of Ca may be initially the result of using some catalysts during the technological process. If there are traces of this element after sliding it is very probable that insoluble products with Ca from water were captured in the superficial layer. Another explanation is less probable: some compounds including Ca migrate from the composite bulk.

Because of false peak of Si, characterising this spectrometry and because glass fibers contain Si, the information may be taken into account in a

qualitative manner, only for pointed out fiber accumulation within the superficial layer. Traces of chlorine are the result of technological process for obtaining PTFE (figure 3 and figure 4). The composite from figure 4 has the greatest variation of the ration between counted atoms of fluor and silicon as it diminishes 2.6 times, comparing the superficial layer after sliding to the initial one. It is obvious that fiber accumulated in the analysed depth of the superficial layer.

Fine traces of Fe show that material transfer is not only one way, from the composite to the metallic surface, but also from the roller to the shoe; fine detached metallic particle are captured by the softer material. (figure 5).

The composite PTFE + 20% glass fiber + 5% graphite is characterised by the same process (diminishing of PTFE concentration in the superficial layer, accumulation of glass fiber as the peak for Si increased and capture of the solid wear debris including steel ones, figure 4).

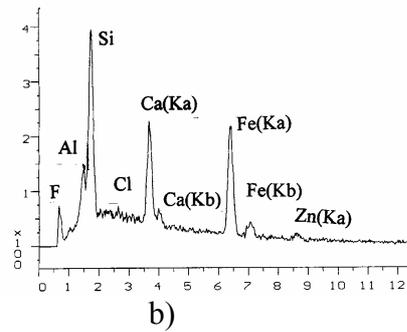
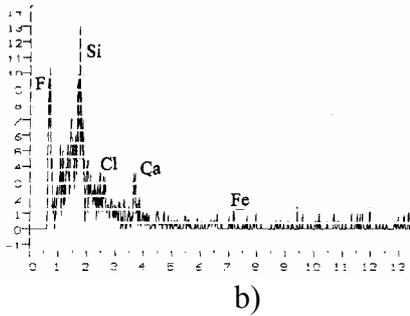
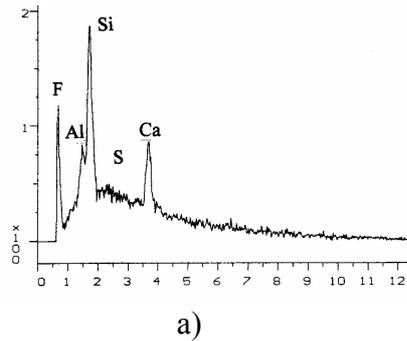
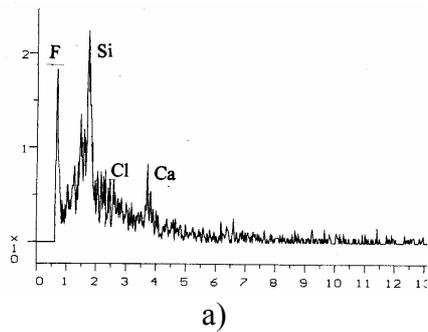
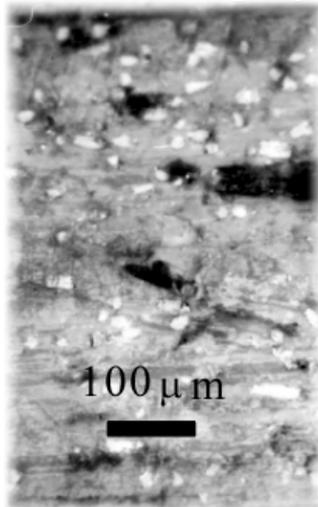
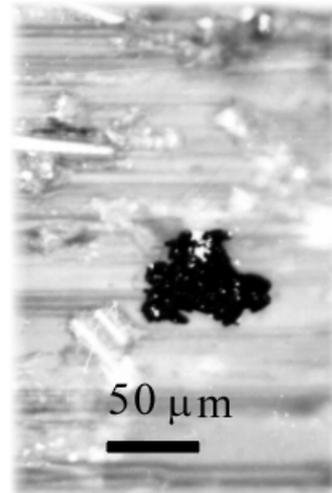


Figure 3 PTFE + 15% glass fiber

Figure 4 PTFE + 20% glass fiber + 5% graphite



a) Water lubrication



b) Dry sliding

Figure 5

## 2. X-RAY DIFFRACTOMETRY FOR THE SUPERFICIAL LAYER

X-ray diffraction is used for studying the crystalline structure, by measuring the symmetry, the dimensions and the imperfections of the crystalline lattice, the size of the coherent scattering domain of the crystalites. The analysis of X-ray diffraction was done by the help of DRON 3 and pointed out qualitatively the modifications within the superficial layer, especially for the glass fibbers. The samples were the same. Investigation depth was  $\sim 80 \mu\text{m}$  and the exposed surface was  $2 \times 10$  (mm x mm).

The crystalline phase characterising the silicon oxide is  $\beta$ -cristobalit.

After sliding in water, one may notice a slight tendency to orient their crystalline phase and to locally finish the structure, possible to explain by forced orientation of glass fiber, obviously from increasing structure peak occurring at an angle  $2\theta=28.26^\circ$  for radiation X-MoK $\alpha$ . The spectra for composite PTFE + 15% glass fiber + 5% MoS<sub>2</sub> is given in figure 6.

Qualitatively, modification of the maximum diffraction intensity  $I_p$  is proportional to the orientation degree of glass fibbers and the changes of diffractogram width in the middle of the spectrum high, noted  $\varepsilon_\beta$ , indicates a structural

change; decreasing of parameter  $\varepsilon_\beta$  indicates a more finished structure.

$$\varepsilon_I = \frac{I_p}{I_{po}} \quad (1)$$

$$\varepsilon_\beta = \frac{\beta_{1/2}}{\beta_{1/2o}} \quad (2)$$

In the above relationships, the index “o” is for the initial value of the respective parameter.

The structure finishing has a mechanical cause for glass fibers, the possible migration of dislocations towards the fiber surface and an increase of the mosaic blocks what are forming the fiber.

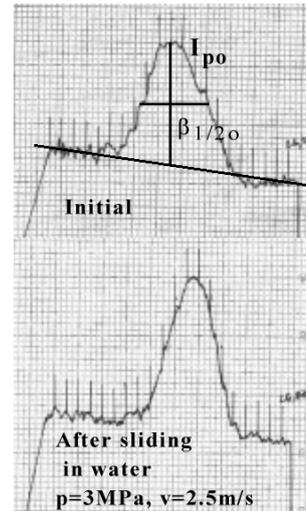


Figure 6.

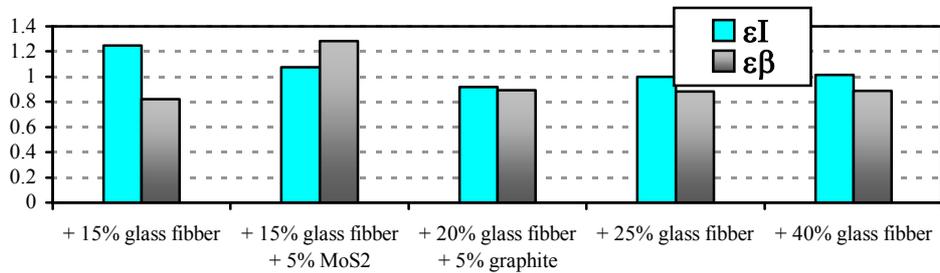
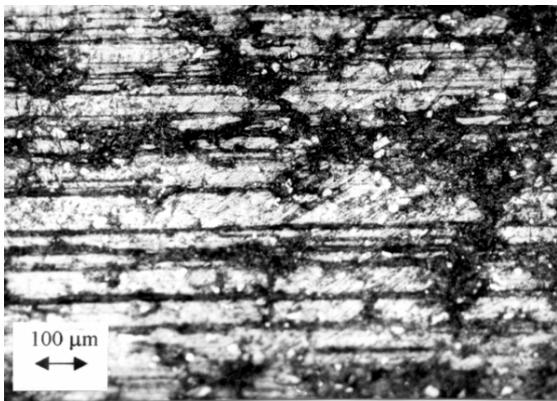
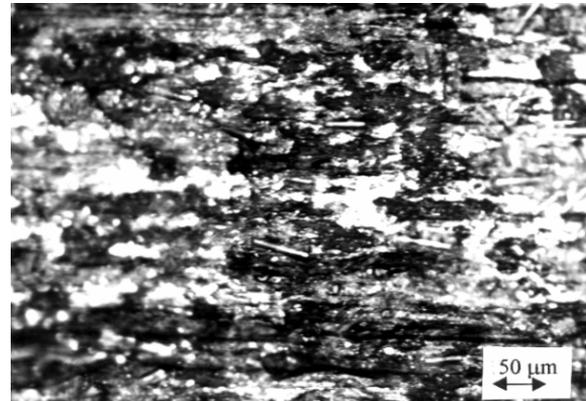


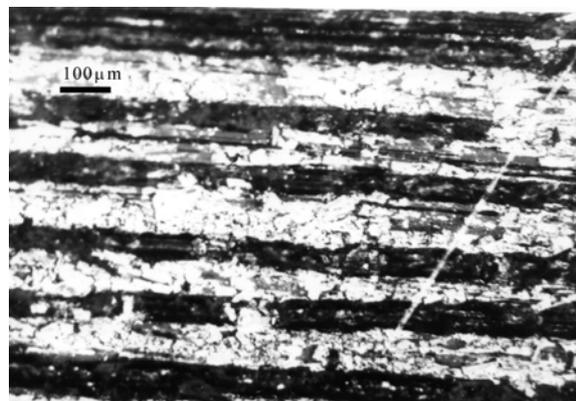
Figure 7



a)



b)



c)

Figure 8

Figure 8 shows the evolution of these parameters for the tested glass fiber + PTFE composites, the initial values being given for comparing reason.

For high glass fiber concentration, the orientation degree is lower and the structure less finished. Thus, as the fiber concentration is increasing, the changes in orientation and structure are less significant. For low concentration (15%), the PTFE matrix is like a soft cushion allowing an embedding of fibers and their orientation. When concentration of fiber increases, the net they create becomes more rigid and it is hard to orient them.

For the composites with MoS<sub>2</sub> and graphite, the modification in orientating and finishing the structure of fibers are smaller, a probable cause might be that these constituents are supporting the load.

The structure is more finished for all composites (figure 7), except the one containing MoS<sub>2</sub>, because water is not favourable to orient and maintain the solid lubricant within the superficial layer. [4, 6]. The fiber orientation is more evident for the composites with a reduced percentage of glass fibers. For the composite PTFE + 20% glass fiber + 5% graphite, it is probable that the graphite does not allow this orientation as it compact the superficial layer.

Microscope study shows that on the composite surface there are fine, partial films of MoS<sub>2</sub>, spread in the sliding direction, but their shape and size depend on testing conditions (figure 8): a) sliding in water in open circuit, v=3m/s and p=4.6MPa, there is the tendency of washing off and oxidation of MoS<sub>2</sub>; b) dry regime at p=0.76MPa and v=1m/s, the thermal field is high and the matrix of PTFE is softening and the bands of MoS<sub>2</sub> fragmentize, mix within the tribolayer, without being favourable to improve tribological behaviour; c) bands of MoS<sub>2</sub>, generated by spreading on the shoe surface similar to the model of lamellar solids [1, 4, 5, 6, 7], for a dry regime less severe: v=0.5m/s and p=0.76MPa, the friction being reduced.

Studies done by Gardos showed that in humid air or in water, MoS<sub>2</sub> oxidises, at least partially, the oxygen atoms replacing the sulphur ones [4]. Thus, in water, the disulphide can not

play its role as a lubricant because it oxidises. But when using a less aggressive lubricant (mineral oil, for instance), an adequate percentage of MoS<sub>2</sub> may improve the tribological behaviour.

Structure is more finished for all tested composites, except the one having MoS<sub>2</sub>, as water is not favourable to maintain the solid lubricant within the superficial layer. [4, 6].

The fiber orientation is more intense for the composite with lower fiber concentration. The values obtained for PTFE + 20% glass fiber + 5% graphite are higher probably because of graphite compacting process in the superficial layer and thus fiber orientation is less obvious.

### 3. CONCLUSIONS

The experimental results on tribological behaviour of the composites with PTFE matrix underline the importance of testing these materials under conditions similar to actual applications. The results recommend the tested composites to be used for water lubrication for an average pressure of: p=0.76...4.6MPa and sliding speed of v=0.5...3m/s. It would be preferred the higher speeds because help to generate a partial or total fluid film and so wear are very much reduced.

In water, wear of composite with PTFE matrix and glass fiber is very low, having a non-linear dependence to the fiber concentration (figure 9). Wear intensity was calculated as

$$I_{wear} = \frac{\Delta m}{\rho \cdot F \cdot l_s} \quad (3)$$

where  $\Delta m$  is the mass loss, under a normal force F and after a sliding distance  $l_s$  (here, of 10500m),  $\rho$  is the relative density of the tested material.

Low variation of investigated X-ray parameters may suggest a greater wear, as the fiber net of the bearing composite is too rigid to become conform to the steel roller. Only laboratory tests similar to actual application may give the optimum percentage of fiber content in the composite. Figure 9 give an approximation by DATA.fit of the experimental results on wear, resulting a better tribological behaviour for a range of 15...25% glass fiber for the tested range 0...40% glass fiber (these limits being characterised by a greater wear).

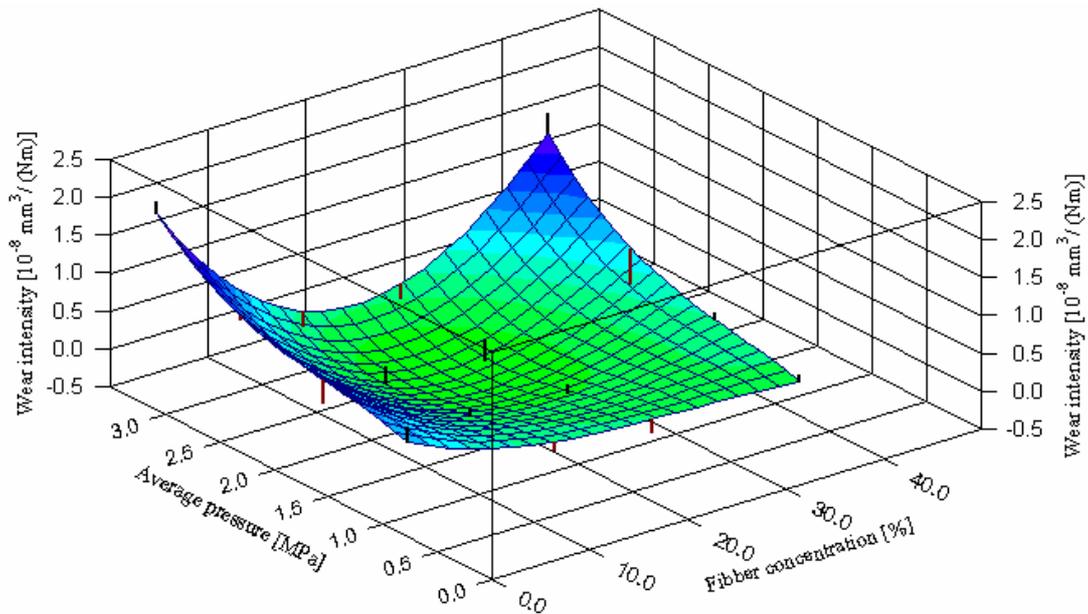


Figure 9. Wear intensity of PTFE + glass fiber composites, after 10500 m of sliding in water at 2.5m/s. [8]

Teflon is not recommended because of its severe wear both for dry and water lubrication condition and its high strain under load. It could be used for bearing supporting a load less than 1MPa.

Adding graphite or MoS<sub>2</sub> does not improve the tribological behaviour when sliding in water but increase the bearing cost. Using these materials as supplementary lubricant in a PTFE matrix composite is necessary only in restricted conditions, for instance when dry regimes are alternated with lubricating ones.

Studies on the tribolayers point out specific processes and could direct the material application in order to optimise the tribological behaviour.

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