Introduction

High performance polymers are used for a lot of industrial applications because of their excellent mechanical properties that are preserved also at high temperatures. Polyoxyethyketone is probably the most attractive among high performance polymers, even if its use is limited by the high cost of supplying and processing and by the high sensitivity to the molding processes. PEEK is generally used as matrix for high performance composites. Tribological components (such as bushes and gears) can be fabricated by injection molding of a PEEK matrix composite, filled with polytetrafluoroethylene (PTFE), graphite particles and carbon microfibers. The maximum amount of filler is generally limited to 30 wt% to avoid problems during injection; PTFE and graphite enhance the tribological behavior of the molded part, whereas the carbon fibers are responsible for the high mechanical performances.

Injection molding is not the only method for making slide-bearing bushes of PEEK composites. In many industrial applications, machining is preferred because of the maintenance and the small-medium scale production series. Injection molded parts are often not allowed to operate due to uneven stress distribution, whereas machined parts tribologically perform better. However, for large scale production series, the injection molding is the only possible choice for bush making and problems related to the lower performances could limit significantly the use of these materials.

In the scientific literature there are a lot of works about the tribology of PEEK, its blends and its composites because of the high industrial interest in these materials. These materials are generally tested under dry sliding conditions [1–4]. Solid particle erosion behavior during sliding [5] and the
correlation between mechanical properties and tribology performance \[6, 7\] were also deeply discussed. Due to the complexity of the problem, statistical techniques \[8\] and neural networks \[9\] were used to study the effect of test parameters on the dry sliding tribological behavior. Moreover, thermal aging can significantly affect the tribological performance of PEEK and short fiber reinforced PEEK composites \[10\].

The investigation on the tribology performance of PEEK, PEEK blends and composites was also extended to water lubricated sliding contacts \[11–15\], different wear modes \[16\] and harsh operating conditions \[17\]. In order to obtain the best tribological behavior, the effect of nano-fillers was discussed (SiC \[18, 19\], alumina \[20–21\] and PTFE \[20\] nano-particles, carbon nanofibers \[22\], nanotubes and nano-onions \[20\]).

The mentioned papers show that PEEK composites have excellent tribological performance among plastics, but the correlation between the injection molding technology and the tribological performance of the molded part has never been investigated. Small laboratory specimens are generally preferred. In 2002, Zsidai et al. deeply discussed advantages and disadvantages of small-scale tribotesting \[23\]. In 2005, Samyn and De Baets studied the friction and wear of acetal, focusing on the effect of the specimen scale on the final measured properties \[24\]. They assessed that to obtain relevant data for practical design of polymer sliding parts, it is necessary to simulate real working conditions as close as possible on laboratory scale. However, they did not discuss that, besides the specimen scale, also the molding process can affect the result of friction and wear tests. In order to avoid scale problems, a purely experimental approach is often used. Plastic gears made of carbon fiber reinforced PEEK \[25–26\] or acetal \[27–28\] were directly tested in working-like conditions. A similar study was never performed on slide-bearing bushes.

In the current work, the authors investigate the capability of a bush made of PEEK composite to substitute a traditional bush made of brass. All the mentioned scientific papers show the high tribological properties of this material, but they do not discuss application cases. Therefore, it is not obvious that injection molded bush behavior under working conditions could be satisfactory.

2. Materials and methods

Commercial bushes were acquired by a manufacturer who started to produce the component depicted in Figure 1 in several high performance polymers. This bush was designed for small size electric motors instead of a traditional brass because of the low weight and production time. The two cavity mold was adapted to injection mold the bush in PEEK 450 FC 30 (by Victrex, USA) which is a 10 wt% carbon fiber, 10 wt% graphite, 10 wt% PTFE filled PEEK. PEEK 450 FC 30 is a commercial material developed for injection molding and investigated in some scientific studies \[4–6, 14\]. At room temperature, it has a tensile modulus of 10.1 GPa, a tensile strength of 134 MPa, a thermal conductivity of 0.78 W·m\(^{-1}\)·°C\(^{-1}\) and a specific heat capacity of 1.8 kJ·kg\(^{-1}\)·°C\(^{-1}\). The manufacturer assured that the molding conditions suggested by the material supplier were observed (mainly the melt temperature of 380°C and the mold temperature of 180°C).

The bushes were used for a short wear test and for material analyses. The wear test was carried out keeping the bush in a standstill position and matching a stainless steel shaft (Figure 2). The shaft was fixed coaxially to the rotor of an electric motor and was put into rotation at 3000 rpm for 10 min. As the shaft diameter was 6 mm, the resulting sliding speed was 0.942 m/s. The bush was mounted on the shaft without interference, therefore a minimum load was present on it in dependence of the weight of the electric motor that was shared by the supports. As an approximation, the load on the bush was about 10 N and the consequent \(pv\) (pressure-speed) value was 0.10 MPa·m/s.

During the test, the bush temperature was acquired by means of a BS1843 standard type \(K\) thermocou-
ple placed on the external surface. A typical temperature curve is reported in Figure 2; after 5 min a plateau is already reached about 55°C, which is relatively low for this material (the continuous mechanical use temperature without impact is 240°C). Ten bush-shaft couples were used in the short wear test. A surface analysis was performed on a bush by means of a scanning surface topography instrument (by Taylor Hobson, Talysurf CLI 2000). As Figure 1 shows, two external lines were chosen for the profile acquisition, the first along the height of the internal cylindrical surface and the second along the height of the external surface. The internal profile was acquired both before and after the short wear test. Also the shaft profile was acquired along the height of the bearing zone.

Some samples were extracted from a bush to carry out DSC tests (by Netzsch DSC 200 PC). In Figure 1 the position of the samples is shown. DSC tests were carried out from room temperature to 400°C at 10°C/min. Other bushes were cut and prepared for optical microscopy.

3. Experimental results

Several bush-shaft couples were used in the short wear test and always the same result was obtained for the shaft: some black bands appeared on the shaft after a few seconds during the test. These bands were not removable by conventional cleaning techniques (such as ultrasounds) but only by machining. The same bushes were used for the microscopical analyses.

The roughness profiles of a bush and a shaft, acquired before the short wear test, are reported in Figure 3. In this figure, the raw data are shown without any filtering and the sample positioning is responsible for the trend. In Table 1 the roughness data extracted from the profiles are collected before and after the test. In Figure 4 the DSC scans performed on 3 samples extracted from the bush are shown. In Figure 5 one of these scans (sample #1 of Figure 1) was compared with two scans performed

<table>
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<th>Bush profile</th>
<th>Shaft</th>
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<tbody>
<tr>
<td>External</td>
<td>Internal</td>
</tr>
<tr>
<td>$R_a$ [μm]</td>
<td>2.53</td>
</tr>
<tr>
<td>$R_t$ [μm]</td>
<td>18</td>
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on bulk PTFE and PEEK that were extracted from commercial bars. In Figure 6 another comparison is shown between two successive DSC scans of the same sample #1. From all the curves of Figures 4–6 both the area of the peak and the temperature of the peak were extracted and reported in Figure 7. The area was calculated after the baseline correction and corresponds to the material melting heat, whereas the temperature is the melting temperature. A double melting peak was observed for all the samples extracted from the bush, therefore two different temperatures are reported for each sample. Figures 8, 9 and 10 show respectively the structure of the molded material in the bulk, the bearing zone and the external surface; in Figure 9 the aspect of the shaft is also superimposed. In conclusion, Figure 11 shows a magnification of the composite structure near the external surface (a) and the bearing zone (b-c).
4. Discussion

During the wear test, the temperature increases up to 55°C in the first 4 min and then it is almost constant. The temperature is low enough to exclude any possible degradation of the material. Nevertheless a strong interaction occurs between the shaft and the bush due to deposition of PEEK composite debris on the shaft surface.

As expected, the acquired profiles of the bush and the shaft are very different from one another (Figure 3). The shaft profile is the smoothest; the bush internal profile is rougher but enormously smoother than the external profile. Comparing data before and after the test (Table 1), the shaft roughness (in terms of $R_a$ and $R_t$) decreases because of the test, whereas the bush inner profile becomes rougher. A small amount of material is transferred from the bush to the shaft; the bush is minimally damaged, therefore its roughness increases; the valleys of the shaft profile are filled with the plastic material and so the shaft roughness decreases. This is a typical phenomenon that can be observed during the sliding of steel on a polymeric surface under unlubricated running conditions. A similar phenomenon was already discussed by Jacobs et al. for the same PEEK composite [14]. A transfer film containing carbon fiber debris can form on the steel counterpart and modify the wear curve. They also discussed that in aqueous environment the formation of the transfer film seems to be disturbed by tribo-corrosion. However, the testing conditions were very different in terms of wear test (a ball on-prism tribometer) and sliding speed (28.2 mm/s), even if the $pv$ term is almost comparable.

DSC analysis allows studying the effect of the injection molding process on the molded material. All the samples present the inflection point related to the PEEK glass transition (at about 150°C) and a double melting peak (Figure 4). The shape of a DSC scan depends, among other things, on the core-skin effect; however the double peak is related to the presence, inside the PEEK matrix, of PTFE which melts at a lower temperature. In fact, in Figure 5 the scan of sample #1 is plotted together with the scan of samples of pure PEEK and pure PTFE. The superposition of the bulk PEEK and PTFE peaks is very similar to the shape of the melting peak of sample #1. Figure 6 shows the comparison between the first and second DSC scans performed on the sample #1. In both scans, two peaks are visible and the difference is negligible. As the first sample melting does not eliminate the presence and the position of the two peaks, these have to be related to the material composition and not to the core-skin effect.

Observing Figure 4, the extent of the PTFE peak is dependent on the amount of PTFE in the sample. PTFE content differs from point to point. This occurrence was never discussed before. The lowest PTFE peak is visible in sample #1, i. e. in the bearing zone. The peak increases in the large rib (sam-
ple #2) and has a maximum in the small rib (sample #3). Actually, the ribs were inserted in the bush geometry only to reproduce the shape of a typical bush made of metal. In fact, many industrial applications deal with the substitution of metallic parts with plastic parts having the same shape, above all for commercial reasons, but also to allow the substitution without any modification in the assembly procedure. A lack of homogeneity in the PTFE distribution can occur because of the complex shape of the bush and other factors, such as the ratio between the viscosities of PEEK and PTFE. Moreover, considering the gate position (Figure 1), during molding carbon fibers are pushed against the bearing zone, rather than toward the ribs. Carbon fibers are present in tribological PEEK mainly to enhance the structural properties; their agglomeration in the bearing zone could be dangerous, PTFE or graphite would be better. DSC data of Figure 7 confirm this statement, as the highest melting heat is measured for sample #3. In fact, melting heat is related to PTFE and PEEK content in the sample, as only these two materials can melt. From Figure 7, it is also clear that the temperatures of the peak are almost constant along the bush, for both materials. Nevertheless, the highest values are observed for the bulk materials, because of the different nature of the polymers.

The injection stage affects the homogeneity of the molded tribological PEEK, as the PTFE, that should help the sliding, is far from the sliding zone, whereas the carbon fibers, that should guarantee the bulk strength, are close to the bearing zone. In order to deepen this aspect, optical microscopy was used to investigate the structure of the molded bushes. In Figure 8 the micrograph of the bulk material is shown: the heterogeneous nature of the PEEK composite is clear. As discussed in [6], the black dots correspond to PTFE; the small shining particles to graphite; the shining filaments to carbon fibers. Due to the injection phase, the carbon fiber orientation is complex. Dealing with PEEK 450 FC 30, previous papers have never investigated the effect of the fiber orientation on transfer film formation and tribological behavior. It is well-known that fibers strongly contribute to the definition of the tribology system but it is hard to understand which is the best orientation distribution. For this reason, testing the real bush in working-like conditions seems to be the only way to investigate its suitability for industrial applications.

In Figure 9 the structure of the composite material near the bearing zone is shown, together with the aspect of the steel shaft after test. After the test, some black parallel bands appear on the shaft due to the transfer film. In Figure 10 the same structure is observed near the external surface. Figures 9 and 10 seem similar, but this similarity disappears after magnification. In the external wall, all the carbon fibers are covered by a thin film of plastic (Figure 11a). In the bearing zone, after a few seconds under working conditions, the same plastic film is removed from the surface by the shaft sliding and the carbon fibers rise (Figures 11b and c). Observing the rising carbon fibers at the internal wall, it is clear that they were leveled by sliding. The transfer film formation damages the bush roughness and alter the aspect of the shaft.

5. Conclusions

In the scientific literature, industrial bushes were never investigated; in this paper, a short wear test was performed on a bush made of composite PEEK. The material showed the expected high properties by limiting the maximum bush temperature reached during the test. The transfer film formation was observed, as well as an increase in the bush roughness. Even if the transfer film could improve the tribological behavior of the bush-shaft system, the presence of debris and the increase of the bush roughness could be negative factors. Moreover, in working conditions, tribo-corrosion could occur in aqueous environment, in dependence of the shaft alloy. These phenomena should be taken into account to evaluate the suitability of the PEEK composite bush for industrial applications. For many industrial applications, it is mandatory that no traces can be left by the bush on the shaft surface. In this case, this kind of material should be avoided. However, even if the deposition of a transfer film is accepted, the molding process of the bush in tribological PEEK should be optimized to maximize the expected performance of the composite; on the contrary, in the industrial practice, the molding process is designed only to obtain the desired geometrical tolerances of the molded part. The heterogeneous nature of the composite material could damage the tribological behavior of the bush; after molding, the PTFE and graphite particles (useful to reduce friction) should be agglomerated near the bearing zone; instead the carbon fibers
should be far enough from there. Injection stage and bush geometry have to be designed carefully. The bush geometry used in this study does not seem to be recommendable for industrial application.

References