Microstructures of tribologically modified surface layers in two-phase alloys

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Abstract. When ductile alloys are subject to sliding wear, small increments of plastic strain accumulate into severe plastic deformation and mechanical alloying of the surface layer. The authors constructed a simple coaxial tribometer, which was used to study this phenomenon in wrought Al-Sn and cast Cu-Mg-Sn alloys. The first class of materials is ductile and consists of two immiscible phases. Tribological modification is observed in the form of a transition zone from virgin material to severely deformed grains. At the surface, mechanical mixing of both phases competes with diffusional unmixing. Vortex flow patterns are typically observed. The experimental Cu-Mg-Sn alloys are ductile for Mg-contents up to 2 wt% and consist of $\alpha$-dendrites with a eutectic consisting of a brittle Cu$_2$Mg-matrix with $\alpha$-particles. In these, the observations are similar to the Al-Sn Alloys. Alloys with 5 wt% Mg are brittle due to the contiguity of the eutectic compound. Nonetheless, under sliding contact, this compound behaves in a ductile manner, showing mechanical mixing of $\alpha$ and Cu$_2$Mg in the top layers and a remarkable transition from a eutectic to cellular microstructure just below, due to severe shear deformation. AFM-observations allow identifying the mechanically homogenized surface layers as a nanocrystalline material with a cell structure associated to the sliding direction.

1. Introduction

Al-Sn alloys have been used as the functional material in journal bearings for compact combustion engines for at least 60 years [1-3]. Fairly little innovation has been observed in this area over the last 50 years. Recently, the authors investigated the need for innovation [4] and, based on the work of Rabinowicz on adhesive wear [5,6], proposed Cu-Mg-Sn alloys as possible new bearing materials with enhanced strength [7,8]. A basic observation in the study of adhesive wear of Cu-Pb and Al-Sn alloys is the formation of a tribologically modified surface layer (tribolayer) [9] which produces severe plastic deformation (SPD) and mechanical alloying (MA) of these two-phase alloys.

The study of tribolayers was pioneered by Rigney and co-workers [10-13], Kapoor [14-17] and Fecht and co-workers [18-20] although an early and important publication in this field is due to Kuhlmann-Wilsdorf [21]. Recently, the interest in this phenomenon has expanded significantly. Several recent studies using copper as a model system [22-29] have improved the understanding of the basic mechanisms in tribolayer formation, while others have expanded the range of practical applications in which the phenomenon is observed, including railway tracks [14-20,30,31], bearings [32,33], brake disks [34,35], medical implants [36] and journal bearings [9].

The study of severe plastic deformation in single phase materials focuses principally on the mechanism of grain refinement by the accumulation of misorientation at subgrain boundaries [37-39], the nature of the grain boundaries [40] and the relationship between grain size and strength [41-44]. Mechanical alloying in two-phase materials has been studied principally in relationship to powder milling processes [45-47]. Early studies on the rolling, wire drawing and ARB of two-phase systems focus on the mechanical properties of the in-situ metal-metal composites obtained in the process [48-54]. Exceptions are the studies on cementite dissolution during wire drawing to high strains of pearlitic steel, where the understanding of the process is essential to explain the extreme...
resistance of this product [55,56]. More recently, strain induced precipitate dissolution [57] and the formation of non-equilibrium structures, including supersaturated solid solutions [58-61] and amorphous phases [61-66] due to SPD has attracted more attention. In the specific case of tribolayers, additional mixing can occur due to the transfer of compounds between the two contacting bodies [67-70], i.e. the formation of a transfer layer. In a previous publication [9], the mechanical modification of journal bearing surfaces was analysed for Cu-Pb and Al-Sn systems under conditions representative of engine operation. While the effect of SPD was clearly demonstrated for both systems, only Cu-Pb presented sufficient evidence to confirm the formation of non-equilibrium solid solutions. In the present paper, a simple experimental set-up will be presented which simulates adhesive wear under the conditions of incipient plastic deformation, as is observed in journal bearings when oil film starvation occurs [9]. The materials tested are a conventional SAE783 (Al 20%Sn 1%Cu) as well as several experimental Cu-Mg-Sn alloys in the cast state. The surface modification is analysed by optical profilometry, SEM and AFM; metallographic sections provide a representation of the microstructural modification of the materials tested.

2. Experiments
To generate the tribolayer, the geometry mentioned in ref. [71] was modified by using a cylindrical pin of 25mm diameter, with a spherical cap of radius 200mm. The spherical cap is brought into contact with the specimen under a load of 100N (Al-based alloys) or 240N (Cu-Mg-Sn alloys) and the pin then rotates around its own axis for 600 sec at 10 cycles/sec, exposing the surface to a wear process which is representative for wear under incipient plastic deformation [71]. The geometry, in the form of a finite-element model, is shown in Fig. 1. It is immediately clear that the contact pressure in the test is heterogeneous. As will be discussed at the end of the paper, several uncertainties exist in the calculations of the friction coefficient \( \mu \); as a consequence, no values of \( \mu \) will be presented here and the work will focus solely on the observed surface modifications.

For Al-Sn alloys, samples were obtained from cast slabs by cold rolling to \( \varepsilon_{VM}=3 \) and annealing at 300°C for 30 min. This treatment provides a close to optimal combination of strength and ductility [72,73]. The same treatment was applied to a commercial hot-rolled slab of AA1100 Al-Alloy, as a reference. The Cu-Mg-Sn samples tested are the same as described in ref. [7], i.e. as-cast material obtained from thin ingots. Their composition is given in table 1.

| Alloy       | Composition (wt%) | Hardness (MPa) |
|-------------|-------------------|----------------|
| Cu-1Mg-1Sn  | 1.05  1.12  0.25  0.01 | 689±72         |
| Cu-1Mg-5Sn  | 0.98  4.90  0.21  ND | 911±162        |
| Cu-5Mg-1Sn  | 5.54  0.81  0.23  0.3 | 2239±207       |
| Cu-5Mg-5Sn  | 5.17  4.34  0.21  ND | 2277±308       |

The surface profile of the wear track was characterized by means of a Nanovea optical profilometer. The wear track was further characterized SEM using a Philips XL 20 microscope with tungsten filament, conventional Eberhard-Thornly SE detector and solid state BSE detector. Selected areas were further investigated with a Bruker Innova AFM in contact mode. Transverse sections trough the center of the wear track were prepared by conventional metallographic techniques and observed unetched in SEM.
Fig. 1. Geometry of the rotating pin, sample holder and load cell of the coaxial tribometer (left). Right: stress distribution in the alloy. The stress distribution is shown in a section through the rotation axis. On the right hand side, a cut-out of the finite element model is presented, with the steel pin (cap radius of 200mm) in contact with a SAE 783 alloy clad onto the steel backing. Notice that the precise stress distribution will be different for each alloy and precise sample configuration.

3. Results
Figures 2. and 3. provide a first impression of the distinct behaviour of the eight materials tested, with the surface profiles of Al-based materials grouped in fig. 2 and the Cu-based materials in Fig. 3. Huge differences are not only observed in terms of roughness but also in terms of the size of the wear track. To keep the measuring time within reasonable limits, only parts of some of the tracks are imaged. Corresponding roughness parameters are summarised in tables 1 and 2.

Some of the SEM-observations are presented in figs. 4 and 5. The AA1100 samples were not analysed in detail, as they merely serve to illustrate the effect of cold deformation and of the addition of Sn to the alloy; they would require special etching techniques to reveal the structure of the tribologically modified layer. In the Al-Sn alloys, due to their two-phase structure, the effects of tribological modification are obvious in the microstructures. For the case of the Cu-Mg-Sn alloys some examples of the materials with 5% Mg are selected, which are highly brittle in tensile tests [7] but behave ductile under the present conditions.

A selected series of AFM-maps, all measured in contact mode, are presented in fig. 6. The maps correspond to the deflection signal and is purposely not traduced into topographic data, as local variations of mechanical properties and chemistry may play an equally important role in the generation of the signal.

Table 1. Maximum and mean square roughness along a 2mm line through the centre of the wear track. CR: cold rolled, ReX: Recrystallised. Notice that, by limiting the determination of the roughness to the centre of the tracks, data can be compared from one material to the other, but the deep grooves observed in cold rolled AA1100 are excluded from the analysis.

| Alloy       | AA1100 CR | AA1100 ReX | SAE783 CR | SAE783 ReX |
|-------------|-----------|------------|-----------|------------|
| $R_{max}$ (µm) | 45.1      | 29.6       | 19.1      | 14.3       |
| $R_{q}$ (µm)   | 9.7       | 6.8        | 3.9       | 3.0        |

Table 2. Maximum roughness and mean square roughness measured along a 2mm track through the centre of the wear track for the four Cu-Mg-Sn alloys.

| Alloy       | Cu-1Mg-1Sn | Cu-5Mg-1Sn | Cu-1Mg-5Sn | Cu-5Mg-5Sn |
|-------------|------------|------------|------------|------------|
| $R_{max}$ (µm) | 10.3       | 6.3        | 14.8       | 7.4        |
| $R_{q}$ (µm)   | 1.8        | 1.7        | 3.5        | 1.6        |
Fig. 2. Wear profiles for the four Al-based samples: a. AA1100 cold rolled, b. AA1100 recrystallised, c. SAE783 cold rolled, d. SAE 783 recrystallised. Notice that in figure 2.a, some of the grooves generated during the test are deeper than the vertical range of the profilometer, which leads to the truncation of the profile. Also important is to consider the horizontal size of the images, which is much larger for the AA1100 Alloys.
Fig. 3. Wear profiles for the four Cu-Mg-Sn samples (as cast): a. Cu-1Mg-1Sn, b. Cu-5Mg-1Sn, c. Cu-1Mg-5Sn, d. Cu-5Mg-5Sn.

Fig. 4. SEM-observations on recrystallised SAE 783. a. presents the border of the wear track. Zone (2) corresponds to the unaffected microstructure which consists of bands of Sn in an Al-matrix. In (1), the two phases cannot be distinguished. In between is a severely deformed transition zone. b. is fully mixed, with some bands of severely deformed, unmixed material.
c. contrasts the original microstructure with severely mixed material (1) and a developing vortex (2). d. shows deformed and fragmented Sn-ribbons underneath a zone of severely mixed material. The different gray tones indicate different Sn-contents, supposedly due to the competition between mechanical mixing and diffusional unmixing.

Fig. 5. As-cast Cu-Mg-Sn alloys. a. shows the border of the wear track in a Cu-5Mg-1Sn alloy. Inside the track, the dendritic structure is still distinguishable. b. shows the fully ductile and completely mixed centre of the same track. c. presents a Cu-5Mg-5Sn alloy. The α-phase (Cu rich solid solution, white) is present as dendrites and as a discontinuous phase in the brittle Cu$_2$Mg matrix of the eutectic zones. In d. this eutectic is transformed into a cellular structure, with a severely mixed, ultrafine structured layer on top.
Fig. 6. a. represents the recrystallised AA1100 surface outside the wear track, b. represents the same alloy inside the track. c. corresponds to the unworn recrystallised SAE783 alloy; d.
is an image of the worn zones, taken in an area where mechanical mixing is complete when observed at SEM-resolution. e. corresponds to an unworn dendrite in the Cu-5Mg-5Sn alloy; f. represents a fully mixed zone inside the wear track. (SD=sliding direction).

4. Discussion

4.1. Use of the coaxial tribometer

The coaxial tribometer used in this work is based on a design used by Stolyarov et al. [71] who consider that this set-up measures the “plastic component” of friction. To the experience of the present authors, it would be better to refer to the “friction at the onset of plastic deformation”. The load in the test is chosen such that the pin produces a very shallow spherical indentation in the material, which grows under the action of the rotating surface. An a-posteriori justification for this method is found in the fact that both the surface characteristics as the microstructure of the material closely resemble what is observed on failed journal bearings retrieved from commercial engines [9].

A basic aspect of the use of this configuration is the determination of the friction coefficient, which is found by integrating Coulomb’s law over the contact circle:

\[ \mu = \frac{2T}{2\pi F} \]

\( T \) and \( F \) are measured with good precision during the test, but the radius of contact varies from alloy to alloy and increases during the test. According to Amontons’ laws [6,74,75], friction is independent of the contact area, but by measuring the torque, the radius inevitably enters into the equation. This also means that the integral must take into account the heterogeneity of the contact pressures along the radius. This heterogeneity is clearly visible in the worn surfaces, with a ring of more severely deformed and mixed material found in the zones of high stresses (Fig. 1). Also, it may be questioned if Amontons’ laws are still valid under the present test condition. The Greenwood-Williamson model [76], as well as recent enhancements of it [77,78], provide ample justification for the mentioned independence, but only cover elastic and partially plastic asperities on the surface. In the present work, large plastic deformation is present and significant modification of the surface topography occurs. Also, in the cold-worked alloys, large grooves are present due to wear, which probably should not be taken into account when integrating the tangential force over the circle.

4.2. Alloy properties and wear behaviour

According to the above, one could consider the present tests as a qualitative tool for ranking different materials with respect to the mechanism of adhesive wear and incipient plastic deformation. The surface roughness and the radius of the wear track are quantitative measures for the amount of wear that occurs in each material.

Considering alloy composition, it is evident that the addition of Sn to Al-alloys dramatically reduces friction and wear, as has been known for many decades [1-3]. Addition of Mg and Sn to Cu-alloys was proposed by the present authors [4] based on the principles established by Rabinowicz [5]. From table 2, it follows that the addition of Mg has a strong effect on wear reduction, while Sn is significantly less effective. The effect of Mg is expected from the tables published by Rabinowicz [5].

Equally important is the effect of cold deformation. In AA1100 and SAE783, the cold-rolled alloys show a much bigger wear track and stronger increase in roughness than the annealed specimens. This agrees with the hypothesis that wear resistance in ductile alloys is associated to the amount of plastic work that can be adsorbed before failure [9], as is justified by the observation that the wear process is basically a manifestation of severe plastic deformation. It must be noted though, that materials which are traditionally considered brittle, such as the Cu2Mg Laves phase in the Cu-Mg-Sn alloys, may show considerable ductility under the high hydrostatic pressures associated to sliding wear [82-84].

4.3. Material flow, SPD and mechanical alloying
The exact mechanism by which the severe plastic deformation is introduced into the surface layer has been a matter of debate. Kapoor [14-17] has proposed a mechanism of ratchetting, i.e. the accumulation of a very high number of small plastic strain increments due to cyclic loading to explain the large strains observed experimentally. Considering the gradual formation of tribolayers and the almost linear increase of the wear track radius observed by the present authors, this explication is most plausible. It does not directly explain the aspect of mechanical alloying and the presence of vortex flow in the microstructures.

Mechanical alloying can be partially explained by the separation of wear flakes from the surface and their re-incorporation at other spots in the material. This would be identical to what is observed in powder milling of multiphase alloys [45-47]. The mechanism is certainly active in the present case and predominates when the load during the test is chosen too high. Under mild loading conditions, it is a secondary mechanism at most. It shall be noted that severe plastic deformation alone is generally sufficient to explain the microstructural alterations of two-phase alloys [48-66].

Tarasov et al. [27,28] have presented interesting models to explain the formation of vortices as a turbulent phenomenon, claiming that the very small length scales produce high Reynolds numbers. One experimental vortex presented by these authors may actually approach the conditions for turbulent flow. The large vortices observed by the present authors (ref. [9], fig.4.c) are difficult to explain by such arguments.

Fig. 4.c actually presents an interesting situation which allows explaining the mechanism of vortex formation and SPD under wear conditions. It is seen how the material is pushed to the left, forming two crests, one which is already severely deformed and mechanically homogenised, while the second one is only starting to rotate. The observed geometry is consistent with chaotic laminar flow [79-81].

4.4. AFM observations

The AFM-images shown in fig. 6 present a consistent pattern for the alloys considered. Outside the wear track, a relatively featureless surface is found, although the AA1100 shows significant patterning, maybe caused by plastic deformation induced by the polishing process previous to wear testing. Inside the track, a fairly regular pattern of cells is found. The images show the magnitude of cantilever deflection at constant contact force; the amplitude of this signal is smaller inside the wear track than outside.

Contact mode AFM is generally considered a straightforward technique to characterise surface topography and in most tribological studies the AFM is used as a high-resolution profilometer. At high resolutions, however, beam deflection is affected by the local compliance of the substrate and by the strength of the interatomic forces between sample and microscope tip [75]. Considering the strong similitude between the observed cell pattern and the subgrain geometry seen in TEM-images, it is concluded that the AFM-observations present the subgrain structure of the modified surfaces and not so much the local topography. Anelastic strain due to the bow-out of dislocation segments in subgrain boundaries and the lower elastic modulus of grain boundaries (due to a less-dense atomic arrangement) would be responsible for a higher cantilever deflection and create a false topography. A few other studies provide analogous observations in worn materials [80] and SPD-processed alloys [85,86]. It is interesting to observe that the cell size found in the severely deformed surface zones of the different alloys follows the general rule that a lower stacking fault energy allows for a smaller grain size in SPD [87-89].

5. Conclusions

The coaxial tribometer test is a useful tool to characterise the adhesive wear behaviour of alloys by means of the surface roughness generated during testing. It is seen how elements which reduce the adhesion tendency with steel, such as Sn in Al-alloys and Mg in Cu-alloys reduce the roughness after testing. Cold work, on the other hand, reduces the deformability of the alloy and therefore significantly increases wear. At the microstructural level, it is seen
how severe plastic deformation is introduced incrementally and leads to a fine mixture of the component phases of the alloys, which can no longer be resolved by SEM. The observed flow patterns and specifically the formation of vortices, suggest that this process can be associated to chaotic laminar flow. AFM-observations in the mechanically homogenised zones of the wear track confirm the nanocrystalline nature of the modified surface.

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