Microstructural and superficial modification in a Cu-Al-Be shape memory alloy due to superficial severe plastic deformation under sliding wear conditions

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Abstract. Stress induced martensitic transformation in copper-based shape memory alloys has been studied mainly in monocrystals. This limits the use of such results for practical applications as most engineering applications use polycrystals. In the present work, a coaxial tribometer developed by the authors was used to characterise the tribological behaviour of polycrystalline Cu-11.5%Al-0.5%Be shape memory alloy in contact with AISI 9840 steel under sliding wear conditions. The surface and microstructure characterization of the worn material was conducted by conventional scanning electron microscopy and atomic force microscopy, while the mechanical properties along the transversal section were measured by means of micro-hardness testing. The tribological behaviour of Cu-Al-Be showed to be optimal under sliding wear conditions since the surface only presented a slight damage consisting in some elongated flakes produced by strong plastic deformation. The combination of the plastically modified surface and the effects of mechanically induced martensitic transformation is well-suited for sliding wear conditions since the modified surface provides the necessary strength to avoid superficial damage while superelasticity associated to martensitic transformation is an additional mechanism which allows absorbing mechanical energy associated to wear phenomena as opposed to conventional ductile alloys where severe plastic deformation affects several tens of micrometres below the surface.

1. Introduction
Shape memory alloys (SMA) exhibit particular mechanical properties represented by the stress-strain curve which for these materials shows a hysteresis loop with a residual deformation during the loading-unloading process performed at constant temperature. This deformation is not produced by dislocation movement but by detwinning of martensite. Therefore, if as effect of increasing temperature, the material transforms from martensite to austenite, the residual deformation could be recovered [1]. In the last decades, this behaviour known as shape memory effect (SME) has increased
the attention in SMA because of the possibility of expanding their use in technological applications. In particular, copper-based SMA has received special attention because of the mechanical properties associated to martensitic transformation (MT) like SME and the super-elasticity effect (SE), understanding that the latter contributes to a damping capability and high fatigue resistance [2,3]. Given the above, both the temperature and external induced stress are factors which play an important role in the phase transformation mechanisms giving origin to SME and SE, this because of the driving force for the MT directly depends of them.

Although Cu-Al SMA has been studied mainly as monocrystals, in recent years several studies have shown important capabilities which make polycrystals of the mentioned alloys multifunctional materials with the possibility to be used in a wide range of applications due to its high thermal stability and a transformation temperature Mₐ which can be controlled by addition of Be [4]. It is known that MT in Cu-Al-Be SMA is a first order diffusionless phase transition induced by stress in which a high temperature face centred cubic (FCC) structure transforms into a low temperature orthorhombic unit cell [5].

Nowadays, some potential applications for SMA are focused in sensor-actuator devices applied for energy recovering [6-8], production of medical instrumental [9], or seismic isolation devices [10-12]. Nevertheless, in the case of Cu-Al-Be SMA, structural applications are limited because of its mechanical behavior is highly influenced by its high anisotropy and large grain size. This issue can be overcome by a grain size refinement produced by severe plastic deformation (SPD) in which a high dislocation density and deformation twins are induced [13].

As it is known, in SPD the increment of dislocation density and subsequent grain fragmentation produces an ultra-fine grain structure (UFGS) with predominantly high angle grain boundaries (HAGB), this refinement process known as continuum dynamic recrystallization (CDRX) increases the material resistance [14-16]. Moreover, in materials like low carbon steel, titanium, copper [17-19], inducing a gradient microstructure by SPD applied in the surface has shown to be a good processing method which improve the tribological performance due to the combination of high strength in the UFG zone and good ductility in coarse grained zone. In addition, a superficial SPD could generate the necessary stress induced martensitic transformation (SIMT) to obtain SE. This combination of SE and high strength in SPD zones could improve the performance of SMA when conducted under sliding contact conditions due to the SE contribute to the dissipation energy during cyclic loads, while high hardness reduces the superficial damage.

In previous works the authors have studied the superficial modification and microstructural layers induced by a gradual SPD process applied in Al-Sn, Cu-Mg-Sn and pure Cu (20, 21). In the present research, a coaxial tribometer developed by the authors (22) was used to characterise the tribological behaviour and microstructural modification of polycrystalline Cu-11.5%Al-0.5%Be SMA in contact with AISI 9840 steel under sliding wear conditions.

2. Experimental method
A cast slab of polycrystalline Cu-11.5%Al-0.5%Be SMA obtained in a high frequency furnace and betatized according to the method proposed in (23) was used for the present work. SMA specimens were thin square coupons with dimensions 20x20x2 mm³ obtained by precision cutting and subsequently prepared by conventional mechanical polishing giving an initial roughness of 230nm±23nm. The superficial SPD was conducted at room temperature using a coaxial tribometer constructed by the authors and described in (22). The SPD process consists in putting a cylindrical pin made of AISI9840 steel into contact with the SMA specimen under a normal load of 400 N. Then, the pin rotates over its own axes during 300s at constant angular speed of 60 rpm. During the test, the normal load was constantly measured with a Burster 8524® load cell, while the torque with a Futek TRS300® sensor.

Worn surfaces were characterized using a Bruker Innova atomic force microscope (AFM) in contact mode and a Phillips XL 20 scanning electron microscope (SEM) equipped with tungsten filament, conventional Eberhart-Thornly secondary electrons detector and solid state backscatter
detector. Transverse sections through the centre of the wear tracks were prepared by conventional mechanical polishing. Microstructural observation of an etched specimen was conducted with SEM using the secondary electrons detector, while a unetched specimen was observed with a Zeiss Axio Imager.A2m reflected light microscope using crossed nicols. Microhardness measurements were made over the transverse section to worn surface using a mechanical Leitz Wetzlar 8068 Vickers indenter.

3. Results and discussion
The diameter of the wear tracks were 1.9mm±0.1mm, with a slight roughness increase in the worn zone of 400nm±20nm. These values suggest a very high wear resistance as compared to previously tested materials like Al-Sn (22) which is commonly used under sliding contact conditions. An important point to take in account for this comparison is that although the wear track diameters are similar and neither of the two materials presented weight loss; the applied load in Cu-Al-Be was four times higher than the applied in Al-Sn, according to the hardness of the former which is also four times higher.

The tribological behaviour of the material can be related with the surface modification shown in figure 1 which corresponds to the SEM image obtained from the worn surface. The elongated flakes aligned to sliding direction are product of the plastic deformation induced during the tribological test and are sign of adhesive wear. A better detail of the zone into the red rectangle is presented in figure 1b, where the pile up of elongated flakes suggest the strain accumulation as described by Kapoor.

Figure 1. SEM images of the worn surface. (a) Elongated flakes aligned to sliding direction, (b) signs of adhesive wear and flakes pile up suggesting plastic ratchetting. (sd=sliding direction).

The surface AFM deflection signal maps inside of the wear track (figure 2) shows a portion of the modified surface in which is possible to distinguish the wear grooves in sliding direction. The shown zone doesn’t present the elongated flakes associated to adhesive wear and plastic deformation. Nevertheless, at higher zoom (figure 2b) is possible to distinguish a regular pattern of cells aligned to sliding direction. The geometry and size of the “cells” are similar to sub-grains observed in transmission electron microscope images, this suggests that the “cells” were generated during the tribological process as effect of the accumulated plastic deformation producing a grain refinement in the closest zone to surface. The most visible patterns in figure 2b correspond to the highest zone of the grooves shown in figure 2a.

Figure 2. AFM maps of the surface inside the wear track. (a) Wear pattern following the sliding direction, (b) Detail over one wear line showing a cellular pattern produced during plastic deformation (notice the difference in scale).
Figure 3 shows the microstructure observed with optical microscope using a light polarizer. In figure 3a, the strong presence of martensite indicates that a SIMT took place during the tribological test, this modified the mechanical properties of the SMA due to the SE behavior in the martensite, giving the material a high damping factor which allows to reduce the effect of the cyclic loads imposed during sliding wear conditions. In figure 3b an arrow points out the fragmented zone of a grain in the surface contact of the material. This fragmentation did not extend through grain; a reason for this could be that the high density of martensitic variants acts as a barrier for the dislocation mobility. The black ellipse indicates martensitic variants bended as effect of the plastic deformation induced at the surface.

![Figure 3](image1.png)

**Figure 3.** Microstructure observed with optical microscope under crossed nicols. (a) General view of the microstructure with coarse grains. (b) Detail of a zone close to surface where is possible to observe the high density of martensite. The arrow points out to the wear-induced structural modification whereas the ellipse shows bended martensitic variants. Sliding direction is perpendicular to image.

SEM images of the zone near the worn surface are shown in figure 4. The martensitic structure is observed in figure 4a; in the top layer (contact surface) is possible to distinguish a discontinuity in the martensitic variants and instead of them, flow lines are visible. These lines are product of the material flow during plastic deformation induced at the surface, In contrast to conventional ductile materials, this zone is confined to a very thin layer.

![Figure 4](image2.png)

**Figure 4.** SEM images of the section transverse to the worn surface. (a) Strong presence of martensite in grains near to surface (b) zoom of the red ellipse zone in which is possible to see flow lines produced by plastic deformation in the top layer.

Hardness results are not shown as a chart because no substantial change was observed through the transverse section, while a slight hardness increment was measured after the surface modification regarding to initial condition. This could be attributed to the SIMT, also is important to notice that because of the MT was carried out homogeneously and the grain fragmentation was experienced only in the top layer, hardness keeps constant through the transverse section.
4. Conclusions

Cu-Al-Be SMA shows a high tribological compatibility when subject to sliding contact conditions against AISI9840 steel. The superficial damage was lower than observed in previous work using the classical tribological Al-Sn alloy, with loads applied which are proportional to the hardness of each material. The improvement of tribological behaviour associated to the high damping factor of SE materials was predicted in previous papers by Schouwenaars and co-workers [25,26].

Contrary to common ductile materials like Al or pure Cu, the grain refinement in Cu-Al-Be SMA was observed only in a very thin layer at the surface, while the expected microstructural gradient was not observed. Nevertheless, a future microstructural characterization of the ultra-fine tribolayer could show a gradual grain fragmentation.

Under the action of the shear deformation imposed during the coaxial tribometry test, a stress induced martensitic transformation was observed throughout the sample. One possible reason for the very limited grain refinement could be that the martensitic variants generated during the transformation act as a barrier for the dislocation mobility and because of this, the continuum dynamic recrystallization associated to obtaining an ultra-fine grain structure does not take place in zones away from the surface.

The combination of the ultra-fine grain structure at the top layer with the SE observed in SMA allows the material to support very high loads under sliding contact without presenting pronounced superficial damage.

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