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Friction and wear of Cu-15 wt%Ni-8 wt%Sn bronze lubricated by grease at room and elevated temperature

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Abstract

Cu-15 wt%Ni-8 wt%Sn (CuNiSn) bronze alloy shows promising bearing performance when used in tribological applications and has attracted increasing research interest. In this work, the tribological performance of CuNiSn in terms of friction and wear were investigated in a ball-on-disc contact configuration, sliding against a Al₂O₃ ball under varying normal loads (1 and 4 N) and environmental temperatures (room temperature (~18°C) and 110 °C). Post-test characterization techniques, including optical profilometry, scanning electron microscopy (SEM), energy dispersive X-ray (EDX) and transmission electron microscopy (TEM) were adopted to characterize the wear track on the CuNiSn surface after 13500 cycles of reciprocating sliding. Both friction and wear behaviour was found to depend on the load and temperature whilst wear resistance reduced with increased temperature. A mechanically mixed layer (MML) and plastic deformation layer (PDL) were characterized by TEM micrographs of the cross-section from the wear track. Under 4 N load, a 1-1.5 µm thick tribolayer was developed during sliding at room temperature compared with a 200-300 nm tribolayer at 110 °C. The friction and wear mechanisms were largely dominated by properties of the tribolayers which were initially associated with and affected by load and temperature.

Keywords: CuNiSn bronze alloy, bearing performance, tribolayer, friction, wear mechanism

1. Introduction

Bronze alloys are widely used as bearing materials due to their favourable tribological performance with or without lubrication in addition to good resistance to shock and impact. Cu-15 wt%Ni-8 wt%Sn (CuNiSn), commercially known as ToughMet 3 (C72900, Materion Corporation), is a newly invented spinodal bronze alloy. The CuNiSn has shown exceptional bearing performance, including lower friction and wear characteristics, improved corrosion resistance, and enhanced toughness, which make the material a promising candidate for demanding applications, such as aircraft landing gear bushings or heavy equipment bearings in aerospace, automotive, oil or gas industries [1-5]. Compared to conventional moderate strength aluminium bronze, CuNiSn is considered to be an improved bearing alloy as it combines high strength and good tribological characteristics. For example, the AT alloy (hot wrought and spinodally hardened CuNiSn) has a 0.2% offset yield strength of 758 MPa that is significantly higher than that of standard complex aluminium bronze alloys AMS 4590, 621MPa. This provides an important potential for weight reduction particularly for the aerospace industry where weight is a critical design parameter [2, 6].

As a modern bearing material, CuNiSn alloy has emerged as a material of choice for bushings or plain bearing components. A systematic mechanical and tribological properties database for CuNiSn is therefore required. Ilango et al. studied the hardness and wear of sand cast CuNiSn (Cu-7 wt%Ni-4 wt%Sn). The Vickers hardness was found to increase with the hardening aging time due to a microstructure change; an adhesive wear mechanism was reported through pin-on-disc wear tests [7].

During sliding of two materials, the high shear force causes plastic deformation at the interacting surfaces, and a transfer layer is commonly formed which correlates with the friction and wear behaviour [8, 9, 10]. Rigney and co-workers studied the structure and chemical composition of the layer for at the

1 contact between a Ni-40wt%Cu block and 440C stainless steel [11, 12]. The transfer layers were found
2 to be a few microns thick and consist of nanocrystalline grains. It was also reported that the loose wear
3 debris was generated from the transfer layer in sliding [11]. Later, by studying the transfer and mixing
4 layer developed between a bronze pin sliding against a brass disc, Rigney concluded that the sliding
5 wear mechanism involves the development of a substructure deformation which leads to transfer,
6 chemical reactions, mechanical mixing and fracture of ductile materials [13]. Godet et al. [14] reported
7 that a transfer layer that is composed of ‘third bodies’ can be created, destroyed and regenerated while
8 the layer flows and transmits load, accommodating velocity gradients.

9 In dry sliding condition, the friction and wear behaviour of CuNiSn alloy has been studied by several
10 researchers [15-19]. Singh et al. [15, 19] assessed the cross-section of CuNiSn in the worn region after
11 dry sliding against 440C stainless steel disc in air and flowing Argon gas through TEM. Distinct
12 nanocrystalline sublayers (mechanically mixed layers, MML) on the pin worn surface were observed
13 and linked to the friction and wear behaviour of the alloy. The top MML was generated due to the
14 compaction and mixing of materials from contacting bodies, which was claimed to improve the wear
15 resistance of the alloy [19]. Cai et al. [16] qualitatively correlated the friction and wear of a CuNiSn
16 alloy pin to the microscopic crystallographic textures and the texture transition in the dry sliding against
17 a bronze disk. Zhang et al. [20] studied the friction of an induction heating manufactured CuNiSn alloy
18 when slid against GCr15 bearing steel lubricated with engine oil, and found that the friction was not
19 affected by the aging time and the wear resistance was enhanced.

20 Although the friction and wear mechanisms of CuNiSn bronze alloy has been widely investigated, these
21 studies were conducted with dry sliding under room temperature. However, in most applications,
22 particularly heavy duty, lubrication of the bronze component is usually required with either oil or grease.
23 In particular, in many tribology systems, the CuNiSn component needs to be operated in a high
24 temperature environment. Nevertheless, the friction and wear mechanism of the CuNiSn bronze alloy
25 under these conditions has yet to be determined. The aims of the study were therefore to investigate the
26 friction and wear properties of the CuNiSn with grease lubrication under both room and elevated
27 temperature, through a ball-on-disc contact configuration and using micro/nano-scopic techniques,
28 including optical microscopy, SEM, EDX, TEM and nano-indenter.

30 **2. Experimental method**

31 **2.1 Wear tests**

32 Reciprocating friction and wear tests using a ball-on-disc configuration and conforming to ASTM G133,
33 were conducted on a UMT-Tribolab testing platform with a REC-400 heating chamber (Bruker
34 Corporation, USA).

35 During the testing, an alumina ceramic ball (Al_2O_3 , from Atlas Ball and Bearing Co. Ltd, UK) with
36 grade 25, a diameter of 4 mm, elastic modulus of 407 GPa, Vickers hardness of 1365 Hv, Poisson ratio
37 of 0.21 was reciprocated against a static wrought and spinodally hardened CuNiSn bronze (ToughMet
38 3 AT 110, from Materion Corporation, USA, henceforth referred to as CuNiSn) disc. The alumina ball
39 was held in a chuck and loaded against the CuNiSn disc. Instead of stainless balls, the alumina ball was
40 used in the present study in order to minimise material transfer onto the testing specimens due to its
41 inert counterface.

42 For CuNiSn sample preparation, 3 mm thick discs were sliced from a diameter of 25.4 mm bar using
43 electrical discharge machining (EDM). In order to obtain identical testing surfaces, each disc was
44 ground with SiC paper from 1200 to 4000 grit following by buff polishing with 1 and 0.25 μm diamond
45 suspensions, leaving a surface roughness of $R_a = 0.2 \mu m$. From this process, at least 200 μm material
46 from the surface was removed in order to get rid of the possible damaged layer caused by the EDM

1 slicing. New specimens (fresh balls and discs) were used for each test and cleaned in an isopropanol
2 ultrasonic bath prior to the test.

3 The reciprocating tests were conducted at both room temperature and elevated temperature of 110 °C
4 and lubricated by AeroShell Grease 64 (5 wt% of MoS₂, Shell PLC, UK). The lubricant grease had a
5 viscosity of 14.2 cSt at 40 °C and 3.4 cSt at 100 °C, and a range of operating temperature from -73°C
6 to +121 °C. The AeroShell Grease 64 was chosen because it is a widely used lubricant in severe
7 operating conditions for which CuNiSn is designed, i.e. aircraft bearing systems. Prior to the sliding
8 test, a thin layer of grease was evenly spread on top of the CuNiSn disc. In the high temperature tests,
9 a REC-400 heating chamber was used to heat up the contact before the sliding test taking place. Test
10 conditions are summarised in Table 1.

11 Table 1. Reciprocating sliding test conditions

Normal load	Stroke distance	Sliding speed	Testing duration	Ball material	Ball diameter	Disc material	Temperature
P = 1, 4 N	4 mm	20 mm/s (2.5 Hz)	90 min (13500 cycles)	Al ₂ O ₃	4 mm	CuNiSn	Room temperature and 110 °C

12 For each test, the total reciprocating cycles were 13500 which produced a sliding distance of 108 m.
13 Based on the material mechanical properties, the initial mean Hertz contact pressures were 582 and 924
14 MPa at P = 1 and 4 N respectively. Each test was repeated once.

15 2.2 Characterization

16 The general morphology and microstructure of the worn surfaces were characterized using an Inspect
17 F FEG scanning electron microscopy (SEM) (FEI, Netherlands) attached with an energy dispersive X-
18 ray (EDX) detector. Dimensions of wear tracks, including 3D morphology, wear scar depth and wear
19 volume loss were assessed using optical microscopy (Alicona InfiniteFocusSL from Alicona Imaging
20 GmbH, Graz, Austria).

21 Focused ion milling (FIB) (FEI Quanta 200 3D SEM/FIB the Netherlands) and transmission electron
22 microscopy (TEM) were undertaken to investigate the cross-section of the worn surface. FIB milling
23 was undertaken using an FEI Helios G4 Nanolab (FEI, the Netherlands). Platinum deposition was
24 applied on the region of interest to prevent Ga⁺ implantation and sputter erosion of the top portion of
25 the surface. TEM observation was obtained using a JEOL JEM-F200 (JEOL, Tokyo, Japan) operated
26 in STEM mode at 200 kV.

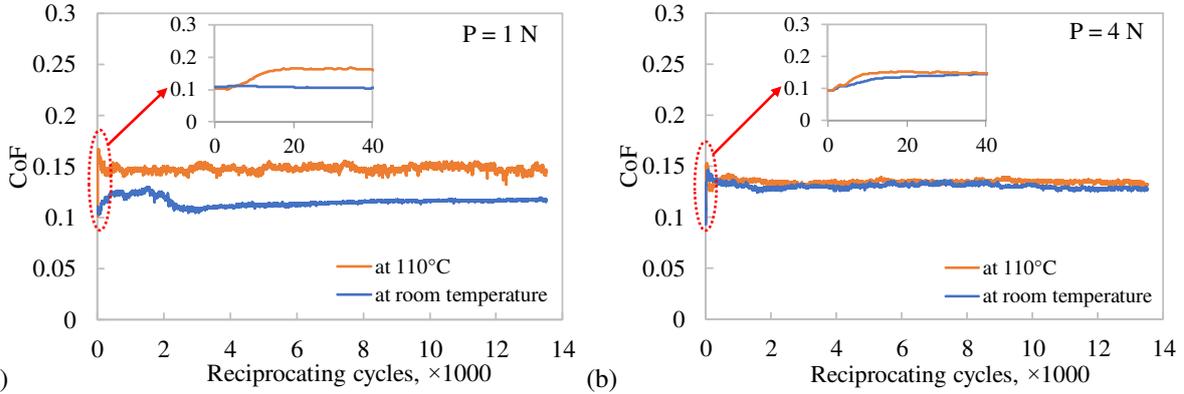
27 Nano indentation (Bruker Hysitron TI Premier, USA) equipped with a standard Berkovich diamond
28 indenter was used for nano-hardness measurement of the worn surfaces. An array of twenty nano
29 indentions was performed at 2 mN load (indentation depth around 110 nm) and intervals of 10 μm
30 between indentations.

31 3 Results and discussion

32 3.1 Friction and wear behaviour

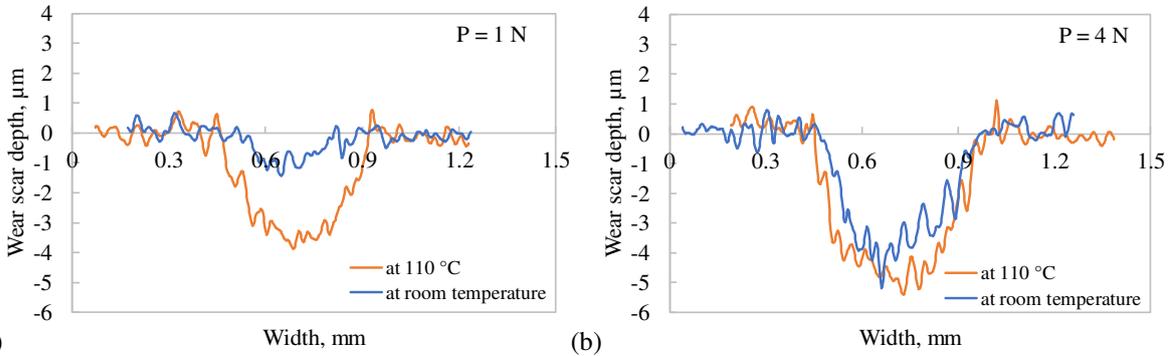
33 Fig. 1 shows coefficient of friction, CoF of CuNiSn evolution with reciprocating cycles for various test
34 conditions. The sliding test showed two stages, a break-in or running-in stage during which CoF was
35 significantly changing, and a steady-state regime in which the average CoF and wear data were roughly
36 constant. The inset figures show the variance of CoF during running-in due to the breakdown of the
37 oxide layer formed on the polished surfaces prior to the test. Apparently, this breakdown was
38 accelerated by higher load and temperature. At P = 4 N, the CoF reaches steady-state rapidly, with
39 comparable values, 0.13 at room temperature and 0.135 at 110 °C. While for P = 1N, the CoF at 110 °C
40 at the steady state was found to be 0.15, which was 1.4 times that at room temperature. Regarding the
41 influence of the normal load, the CoF was found to be increased from 0.11 under 1 N to 0.13 under 4
42 N at room temperature. At 110 °C, the CoF decreased with load, from 0.15 to 0.135 which was
43 comparable to that at room temperature. This indicated that the interface that governed the frictional

1 behaviour of the countering materials may have different properties caused by varying operating
 2 parameters, i.e. load, temperature. Post-test analysis towards the wear track, described later, provides
 3 further information.



4 (a) (b)
 5 Fig. 1 CoF between alumina ball and CuNiSn disc under loads of (a) $P = 1\text{ N}$ and (b) $P = 4\text{ N}$, at both room
 6 temperature and $110\text{ }^\circ\text{C}$

7 After the wear test, the CuNiSn disc and the countering alumina ball were cleaned using isopropanol to
 8 remove remaining grease for optical and electron microscopy. Three-dimensional wear scar profiles
 9 were measured using the Alicona microscope for quantifying the wear volume. The cross-section profile
 10 of the worn surface perpendicular to the sliding direction is shown in Fig. 2. It is evident that the wear
 11 of CuNiSn was influenced by both load and temperature. Higher load and temperature resulted in severe
 12 operating conditions which deteriorated the interface between the CuNiSn disc and the alumina ball.
 13 The largest wear scar was produced under 4N and elevated temperature, as shown in Fig. 2 (b). After
 14 cleaning there was no surface damage or wear observed on the alumina ball by using the optical
 15 microscope.



16 (a) (b)
 17 Fig. 2 Wear scar profiles on the CuNiSn discs measured by Alicona for (a) $P=1\text{ N}$ and (b) $P=4\text{ N}$, at both room
 18 temperature and $110\text{ }^\circ\text{C}$

19 Specific wear rates W , in $\mu\text{m}^3/\text{Nm}$, were calculated using:

$$20 \quad W = \frac{V}{Pvt} \quad (1)$$

21 Where V is the wear volume loss, in μm^3 , P is the normal load, in N , and v is the sliding speed, in m/s
 22 and t is the test duration, in s .

23 Resulting CoF, wear scar dimensions and specific wear rate are summarised in Table 2. The specific
 24 wear rate was found to increase with load at room temperature but decrease at elevated temperature.
 25 For all the conditions considered, the lowest specific wear rate was found to be $0.27 \times 10^4 \mu\text{m}^3/\text{Nm}$ at
 26 room temperature while the highest was found to be $1.53 \times 10^4 \mu\text{m}^3/\text{Nm}$ at $110\text{ }^\circ\text{C}$, both of which
 27 occurred at $P = 1\text{ N}$. This agrees with the CoF values from the same testing conditions. The results
 28 indicated that under the moderate load, the temperature affected the interface dominantly. This is

1 because the grease viscosity and the temperature are inversely proportional, i.e. the higher the
 2 temperature the lower the viscosity and hence the thinner the lubricating film. Consequently, friction
 3 was found to be higher at $P = 1$ N, $110\text{ }^{\circ}\text{C}$, as shown in Fig. 1 (a). In contrast, under higher load, $P = 4$
 4 N, a lubricant layer was hardly formed at the interface no matter what the temperature was. In this case,
 5 the interface was mainly influenced by the load. Consequently, the friction (Fig 1. (b)) and wear scar
 6 profile (Fig 2. (b)) showed little difference between the two testing temperatures.

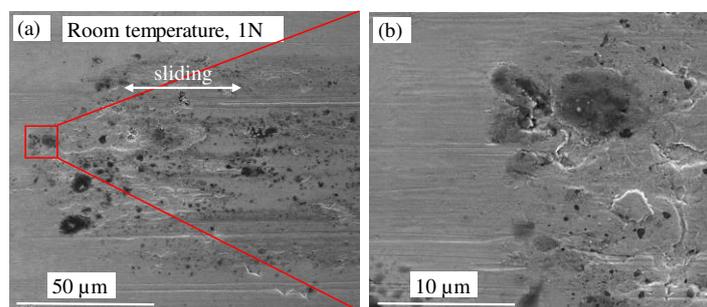
7 Table 2. Wear track dimensions and specific wear rate for varying testing conditions

Environment	Coefficient of friction	Wear scar width, μm	Wear scar depth, μm	Wear volume, $\mu\text{m}^3, \times 10^6$	Specific wear rate, $\mu\text{m}^3/\text{Nm}, \times 10^4$
Room temp, 1N	0.11	300	1.4	0.29	0.27
Room temp, 4N	0.13	590	5.2	2.02	0.47
$110\text{ }^{\circ}\text{C}$, 1N	0.15	480	3.9	1.65	1.53
$110\text{ }^{\circ}\text{C}$, 4N	0.134	600	5.4	2.72	0.63

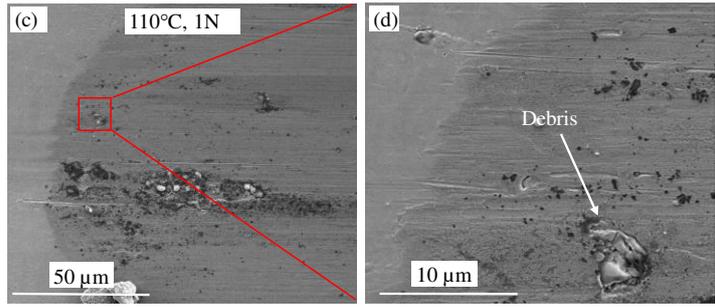
8 3.2 SEM characterization of the worn surface

9 To evaluate the friction and wear mechanism between the CuNiSn disc and alumina ball, morphology
 10 and microstructure of the wear tracks were further characterized post mortem. Fig. 3-6 show SEM
 11 images of wear tracks on the CuNiSn disc after 13500 cycles of rubbing under varying loads and
 12 temperatures. Prior to SEM examination, the CuNiSn disc surface was ultrasonically cleaned with
 13 isopropanol. As the characterization of the used grease was not considered in the current study, the
 14 grease mixed with wear debris was removed prior to the cleaning process.

15 The SEM micrographs show that the microstructure of the wear track varied along its length, i.e. from
 16 the track end to the mid zone of the track. During sliding, transferred material was pushed to the side
 17 of the track mainly in the sliding direction, similar to snow shovelling. At the track end reversals
 18 transferred material accumulated. In addition, from Fig. 3 and 4 it can also be seen that the morphologies
 19 of worn surfaces were significantly different under the two testing temperatures. At $110\text{ }^{\circ}\text{C}$, wear debris
 20 was accumulated at the end of the track (Fig. 3 (c)), while under room temperature, sparse oxidation
 21 zones (dark regions in Fig. 3 (a) and (b)) were observed on the worn surface. Wear debris in Fig (3)
 22 showed evidence of more material transfer caused by plastic deformation of the surface material.
 23 Moreover, at the middle of the wear track (Fig.4), fine uniform wear furrows were seen on the wear
 24 track rubbed under $110\text{ }^{\circ}\text{C}$ (Fig. 4 (b)) whilst a large sized groove was observed at room temperature.
 25 This indicated that temperature played an important role on the contact interface properties, and that the
 26 abrasion of wear debris, working as ‘third body’, was the main wear mechanism for the tests under 1N
 27 and room temperature.



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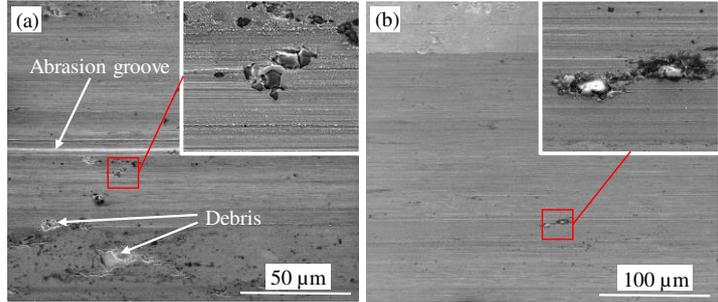


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Fig. 3 SEM images showing the top view of the end-stroke wear tracks and detailed microstructure at $P = 1N$, and (a) & (b) at room temperature, (c) & (d) at 110 °C



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Fig. 4 SEM images showing the top view of the mid-stroke wear tracks and detailed microstructure at $P = 1N$, and (a) at room temperature, (b) at 110 °C

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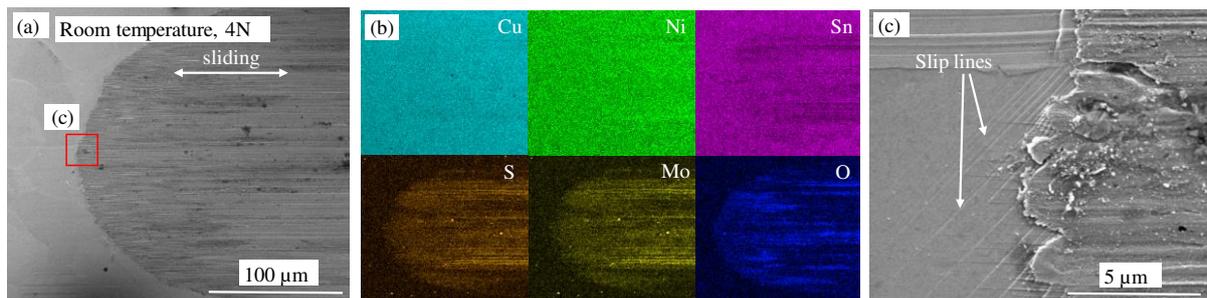
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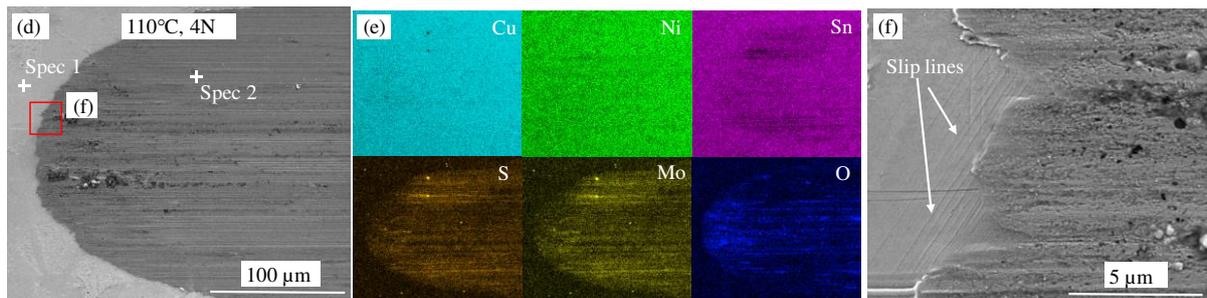
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16

Fig. 5 and 6 show the microstructure and chemical composition of wear tracks subjected to $P = 4N$ and varying temperature. It is apparent that higher load produced larger sized wear tracks and the abrasive wear mechanism dominated. Slip lines were observed on the un-worn surface near the edge of the wear track, as shown in Fig. 5 (c) and (f). This was due to the synergistic effects of the high normal and shear stresses occurred at the interface. Detailed composition information for specs 1 and 2 on Fig. 5(d) are shown in Tables 3. According to the EDX maps and point analysis shown in Fig. 5(b), (e), and Table 3, the wear track was predominantly covered by an oxide layer. Bronze alloy elements Cu, Ni and Sn were found to be distributed over both fresh and worn surfaces of the CuNiSn disc. Distribution of S and Mo elements from solid lubricant additives in grease were found to be distinguished from fresh surface which were mechanically mixed in the top layer on the wear track.

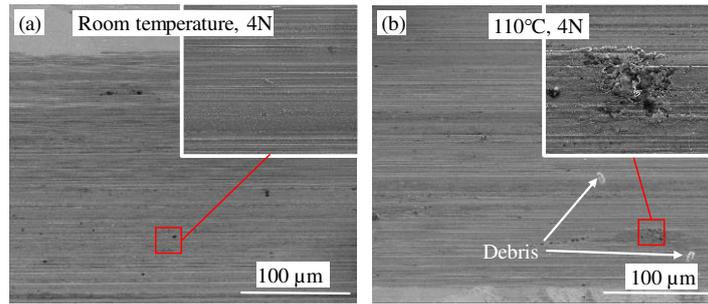


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1 Fig. 5 SEM images showing the microstructure and EDX mapping of the chemical elements distribution for the
 2 end-stroke wear track at $P = 4$ N, (a) (b) (c) at room temperature, and (d) (e) (f) at 100°C



3
 4 Fig. 6 SEM image showing the mid-stroke wear track and the microstructure at $P = 4$ N, (a) at room
 5 temperature, and (b) at 100°C

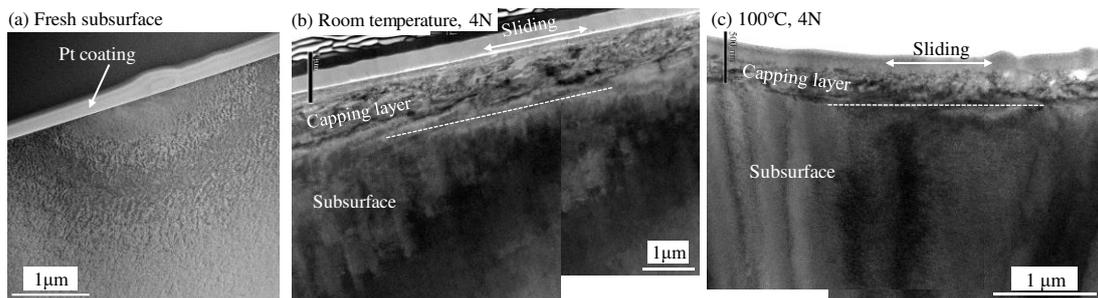
6 Table 3. EDX point analysis of chemical composition (At %) for points Spec 1 and 2 (shown in Fig. 5 (d))

Point	Cu	Ni	Sn	C	O	S	Mo
Spec 1	54.9	12	2.9	29.1	1.1	-	-
Spec 2	50	11	2.4	32.7	3	0.7	0.3

7 3.3 TEM characterization

8 While SEM characterization shows the micro-structure of the worn surface from the top, a cross-section
 9 analysis yields depth information for subsurface material deformation and formation of the transfer
 10 layer, which is critical to reveal the wear mechanism.

11 Fig. 7 shows the TEM micrographs of the wear track cross-section on the CuNiSn disc after 13500
 12 cycles at both room temperature and 110°C . Compared to the fresh subsurface (Fig.7 (a)), an influenced
 13 layer right beneath the Pt coating were observed capping on the wear track at both test temperatures,
 14 which is known as a tribolayer. The thickness of the capping layer generated at room temperature was
 15 $1\text{-}1.5\ \mu\text{m}$ (Fig.7 (b)) compared with $200\text{-}300\ \text{nm}$ for 110°C (Fig.7 (c)). A deformation substructure
 16 was seen on the cross-section for both testing temperatures after 13500 cycles. A similar deformation layer
 17 was observed in a previous study by Singh et al. [15], in which a CuNiSn bronze pin was slid against a
 18 stainless steel disc without lubrication and under room temperature. The capping layer formation
 19 process involves deformation, transfer, chemical reaction, and mechanical mixing [21], and therefore
 20 determined the friction and wear behaviours of the CuNiSn.

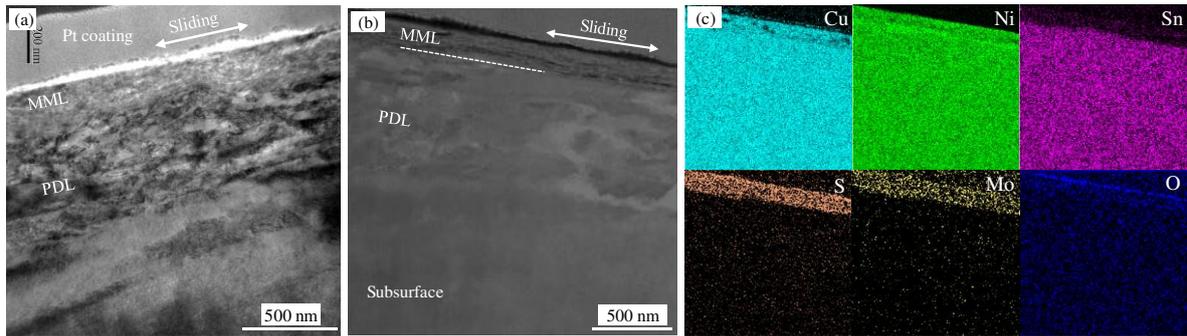


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 22 Fig. 7 Bright field cross-section TEM micrographs, (a) fresh subsurface, (b) and (c) formation of a capping layer
 23 in the immediate vicinity of the surface at room temperature and at 100°C testing temperatures respectively

24 Fig.8 (a) shows the bright field cross-section microscopy of the CuNiSn wear track and provides
 25 tribolayer details at room temperature and $P = 4$ N. Two sublayers beneath the surface within the
 26 tribolayer were observed, as shown in Fig. 8 (b). The top layer close to the surface is a thinner, fine-
 27 grained mixed material that is denoted as a mechanically mixed layer (MML), with a thickness of
 28 approximately $200\ \text{nm}$ (Fig. 8(b)). It is reported that this layer has a different composition from that of
 29 the bulk material [21, 22, 14]. This MML separated the direct contact between CuNiSn and the alumina
 30 ball, transmitting load and accommodating the shear velocity gradients along the interface. It was

1 generated during sliding and could be destroyed and regenerated [14]. Fig.8 (c) shows the EDX mapping
 2 of Fig. 8 (b), revealing the chemical composition of the MML. Chemical elements S and Mo were from
 3 the additives in the lubricant grease. The environmental source, lubricant grease in this work became a
 4 source of chemical species that were incorporated in the MML. An even thinner O-rich layer was
 5 observed stacking on top of the MML, shown as a narrow dark zone in Fig. 8 (b). This may be due to
 6 metal oxidation occurring on the worn surface in the post-test cleaning process. During the sliding test,
 7 the CuNiSn surface was covered with grease, shielded from the reactive environment, so the oxidation
 8 process was prevented. The plastically deformed layer (PDL) was right below the MML in the base
 9 material. A clear demarcation was observed between the deformation substructure of PDL and the
 10 MML, shown in dark field cross-section image (Fig. 8 (b)). A fine-grained sublayer PDL was made of
 11 nanograins elongated along the sliding direction. By comparison TEM examination, Singh et al.
 12 reported that the grains were heavily deformed, indicating large strain had been experienced by the
 13 sublayer during the sliding wear test [15]

14

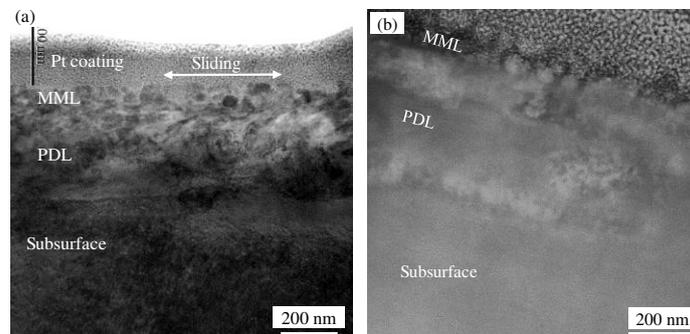


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16 Fig. 8 TEM micrographs showing details of the capping layer at room temperature and $P = 4$ N, (a) bright field
 17 cross-section (b) dark field cross-section in the adjacent region of (a), and (c) EDX mapping showing the
 18 chemical elements distribution in (b)

19 Fig.9 shows the bright and dark field microscopies of the cross-section of the wear track under 110°C
 20 and $P = 4$ N. In contrast to the room temperature case, the MML was barely observed in the TEM
 21 micrographs as there was no boundary presented against the PDL. The overall deformed layer was much
 22 smaller than that under room temperature (Fig. 8). It can be concluded that the tribolayer, especially
 23 the MML was hardly developed or easily destroyed on the wear track during sliding at 110°C . As the
 24 behaviour of this layer depends on the dimensions, compositions and properties, it can be beneficial or
 25 harmful to the friction and wear of the material. Indeed, as shown in Fig.1-2 and Table 2, CuNiSn
 26 demonstrated lower friction and higher wear resistance at room temperature than at 110°C . This is
 27 probably due to the fact that the tribolayer was more efficient in reducing friction and wear at room
 28 temperature than 110°C for the CuNiSn and Al_2O_3 tribolo-system.

29



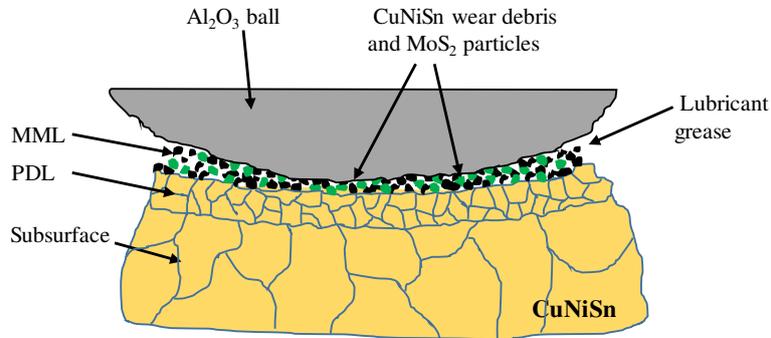
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31 Fig. 9 TEM micrographs showing details of the capping layer at 110°C and $P = 4$ N, (a) bright field cross-
 32 section (b) dark field cross-section in the adjacent region of (a)

33 Fig. 10 shows the schematic diagram of the tribolayer in the sliding contact between the alumina ball
 34 and the CuNiSn. The operating conditions, temperature, load and lubricant, determined the formation
 35 and rupture of the tribolayer. The lubricating grease helped to separate the contacting surfaces, and

1 introduced the solid lubricant MoS₂ particles which contributed to friction and wear reduction. In
2 reciprocating rubbing, abrasive wear debris from the CuNiSn together with the MoS₂ particles were
3 mechanically mixed and stacked on top of the plastic deformed layers, such as MML and PDL shown
4 in Fig. 10. Therefore, it is concluded that the friction and wear of the CuNiSn bronze is largely
5 influenced by the formation/rupture of the tribolayer.

6



7

8 Fig. 10 Schematic diagram of the tribolayer between alumina ball and CuNiSn bronze under sliding friction

9 3.4 Nano-indentation hardness

10 To study the mechanical property of the tribolayer generated on the CuNiSn surface, the nano-hardness
11 was measured with a Bruker's Hysitron TI Premier Nanoindenter. Twenty nano-indentations were
12 performed along the central line of each wear track, as shown in Fig. 11 (a). The interval between
13 indents was 10 μm so a total length of 190 μm of the wear track was measured. The instrumented
14 indentation method built in the nano-indenter provided a continuous record of the variation of
15 indentation load as a function of the penetration depth. Fig. 11 (b) shows the typical load-depth curves
16 for varying testing conditions. From the load-depth curve, the individual hardness of each indent was
17 analysed using the Oliver and Pharr method [23, 24], which was incorporated in the analysis program
18 on the nano-indenter. The average hardness from the twenty indents is shown in Fig. 11 (c).

19 From Fig.11 (b) it can be seen that the indentation depth for different testing conditions varied from
20 105 to 115 nm. These depths were comparable to the thickness of the MML on the wear track from the
21 TEM micrograph (Fig.8 and 9) and so relate to the properties of the MML and not the substrate. Worn
22 surfaces had a lower nano-hardness compared with the fresh one. At 1N load, after 13500 cycles, the
23 surface hardness of the worn surface showed an approximate 8% reduction compared to that of the
24 unworn surface. At room temperature, the nano-hardness was 5.05 GPa at $P = 1\text{N}$ and 4.77 GPa at $P =$
25 4N , compared to 4.97 GPa and 4.74 GPa at 110 °C. In sliding, the unworn CuNiSn surface with higher
26 hardness was deteriorated and incorporated with material transfer and mechanical mix. Apparently, the
27 top layer composed of loose particles and debris demonstrated a lower nano-hardness at both test
28 temperatures. However, a slightly higher nano-hardness under room temperature was associated with a
29 better wear resistance compared to that at 110 °C (lower specific wear rate in table 2). The mixed
30 material on the surface, seen as elevated plateaux, softer than the fresh surface material, played an
31 important role in determining the wear property of CuNiSn. Due to the limited nano-indentation depth,
32 there is no indicative parameter that can be used to analyse the mechanical property from the lower
33 layer, PDL, or even deeper to the subsurface of the bulk material. However, as the microstructure size
34 was found to be decreased in PDL, it suggested that the micro-hardness in this layer was greater than
35 with the subsurface, similar to the process of mechanical hardening [25, 26].

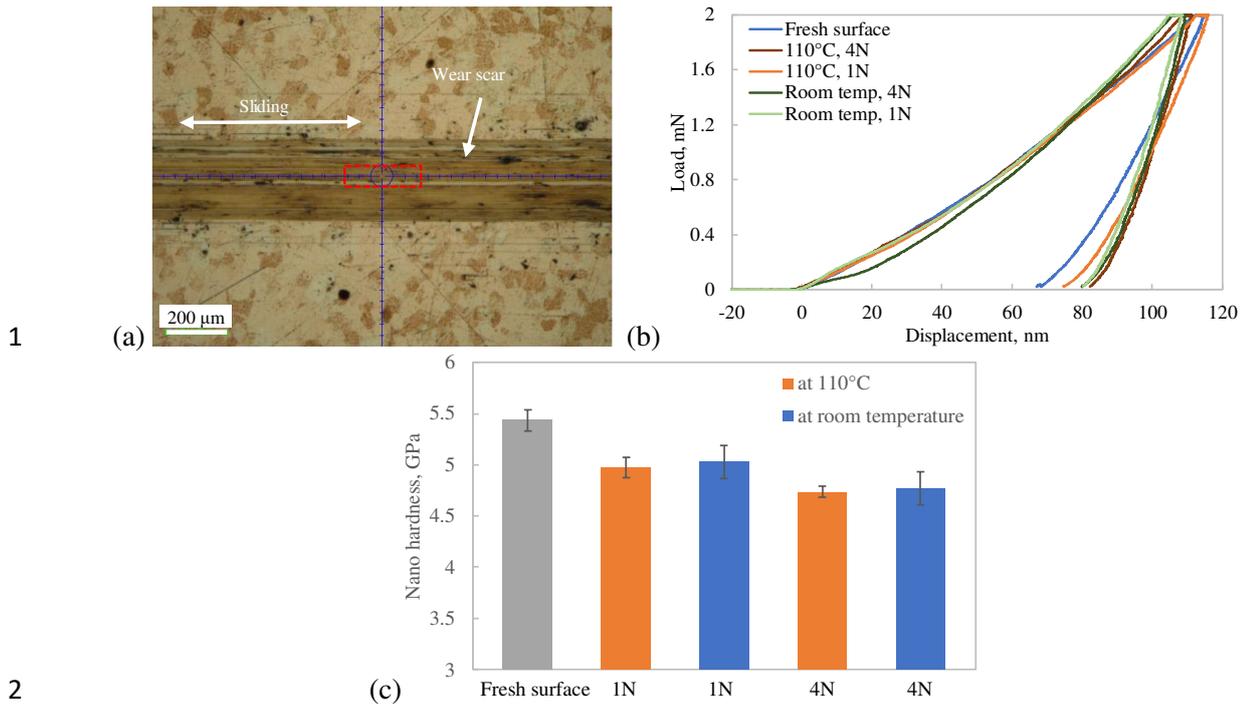


Fig. 11 (a) Location of nano-indentations, (b) typical nano-indentation load-depth curves for varying conditions (one curve selected from 20 for each testing condition), and (c) Nano-hardness of wear track on the CuNiSn bronze

4 Conclusions

CuNiSn bronze was experimentally studied using a ball-on-disc reciprocating sliding configuration against alumina ball under varying operating conditions and environments. Friction and wear resistance were analysed using post-test techniques, including Alicona optical microscopy, SEM, TEM and nano-indenter. Conclusions from the study are summarized as follows:

- The coefficient of friction, CoF, of CuNiSn bronze was influenced by the environmental temperature. Under 1N normal load, the CoF at the steady stage was 0.15 at 110 °C compared to 0.11 at room temperature. Both the load and temperature had significant influences on the bronze's CoF.
- The wear resistance of CuNiSn reduced at elevated temperature. Under 1N load, the specific wear rate was $1.53 \times 10^4 \mu\text{m}^3/\text{Nm}$ at 110 °C compared with $0.27 \times 10^4 \mu\text{m}^3/\text{Nm}$ at room temperature.
- The wear mechanisms of CuNiSn against the alumina ball were investigated with SEM characterization. The chemical elements S and Mo from the grease additives were found to be mechanically mixed in the top layer of the wear track.
- Microscopic view of the cross-section for the CuNiSn wear track was measured with a TEM. The tribolayer of deformed material beneath the wear track surface was found to be 1-1.5 μm at room temperature and 200-300 nm at 110 °C. Two sublayers, MML and PDL were observed with different structures and compositions.
- Nano hardness of the wear track on the worn CuNiSn surface decreased by 8% compared to that on the fresh surface. The EDX mapping confirmed that the top layer on the wear track was composed of wear debris, oxidation and lubricant materials that were mechanically mixed and layered on the wear track. This tribolayer governs the friction and wear of CuNiSn bronze in sliding against an alumina counterface.

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5

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