THE POTENTIAL OF INDIUM AS A SOFT METAL LUBRICANT REPLACEMENT FOR LEAD

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ABSTRACT

A review was carried out comparing the properties of several soft metals to identify the most suitable lubricant alternative to lead. Indium emerged as the most promising candidate.

A method for producing thin-film indium coatings via physical vapour deposition was developed. Coatings were deposited onto 52100-steel discs and bearing balls and assessed for their adhesion, purity and morphology. The friction and lifetime were then assessed in vacuum through spiral orbit tribometry (SOT) and the tribological performance compared with that of lead and MoS_2.

The indium coatings displayed a microstructure consisting of evenly distributed nodules of pure indium. Coatings on bearing-steel discs exhibited both good adhesion and high chemical purity. However on bearing-steel balls, adhesion was weaker.

During SOT testing indium transferred readily from the coated ball to the uncoated SOT plates. The coatings displayed low friction (lower than for lead and comparable to that of MoS_2). Whilst indium coating lifetimes exceeded those of MoS_2, they were appreciably shorter than those observed with lead coatings of similar thickness.

1. INTRODUCTION

1.1. Background

Lead lubrication is commonly used on precision bearings in European spacecraft mechanisms. It is often chosen because it has been demonstrated that ball bearings coated with sputtered (and ion-plated) lead, and fitted with leaded bronze cages, exhibit very long lives (up to 10^9 revs) in vacuum. This longevity has also been shown using a spiral orbit tribometer (SOT), where the long lifetime was attributed to the ductile nature of the soft metal allowing ready transfer and re-distribution over all contact surfaces [1].

However, lead is a toxic material and in recent years its use has been banned in several applications under the EU Restriction on the Use of Hazardous Substances (RoHS). At the time of writing the use of lead coatings in equipment designed for use in space is acceptable, but there is an implication that on-ground usage of such equipment will eventually fall under the ban. Other lead-containing components used in space mechanisms (e.g. leaded-bronze cages) have been made exempt by the RoHS authorities. In conclusion, the legislation as applied to lead-lubricated bearings is ambiguous, but, whilst there is no immediate threat to their use in space applications, it is our belief that eventually the legislation will encompass this area. In anticipation of the need to find a replacement lubricant we have attempted in this study to identify and assess a suitable alternative.

1.1. Review of Potential Soft Metal Lead Replacements

A review of potential replacements for lead-lubrication [2] considered the lubricating properties of several soft metals (gold, silver, platinum, indium and tin). Tab. 1 summarises the properties of these metals and compares them to lead.

Consideration of the information provided in Tab. 1 would suggest that all five candidate coatings meet the criteria regarding vacuum compatibility, melting point and lack of toxicity, and that all (although these are not essential requirements) are reasonably good conductors of electricity and heat.

Fig. 1 compares the frictional properties of the soft metals (except Pt) listed in Tab. 1.

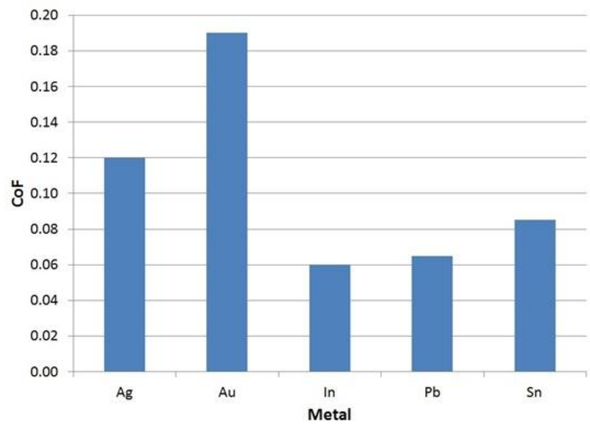

Figure 1. Friction coefficients of soft metal films in vacuum at room temperature (pin Si_3N_4, disc 440C, load 10N, speed 24mm/s) [3].

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Table 1. Properties of potential soft metal replacements to lead [2].

Soft Metal	Shear Strength (MN/m^2)	Hardness (mhos)	Melting Point (°C)	Order of Chemical Reactivity (1 = Least Reactive)	Temperature (K) for VP > 10^{-13} mbar	Thermal Conductivity (W/m.K)	Electrical Resistivity at 273K (nΩm)	Toxicity
Lead, Pb	26.5	1.5	327.5	5	~475	35.3	192	Toxic
Gold, Au	60	2.5	1064.4	1 (inert)	~900	318	21	Non-toxic
Silver, Ag	37.8	2.5	961.8	3	~600	429	15	Non-toxic
Platinum, Pt	-	3.5	1768.4	2	~1200	71.6	96	Non-toxic
Indium, In	11.8	1.2	156.6	4	~600	81.8	80	Non-toxic
Tin, Sn	12.6	1.5	231.9	6	~750	66.8	115	Non-toxic

Platinum has received little attention as a lubricant and consequently there is little documented evidence on its performance as a thin lubricating film. However, some pin-on-disc tests carried out in air on 0.6μm thick platinum coatings applied to bearing and high-speed (M2) steels would indicate friction coefficients in the range 0.2 to 0.3 [4]. Although the test conditions were not identical to those of Fig. 1, they were sufficiently close to suggest that platinum's frictional behaviour is closer to that of gold than of other soft metals. Therefore, from the known frictional properties of these soft metal coatings it would appear that both gold and platinum yield appreciably higher friction than lead. As such, both gold and platinum were eliminated as lead-replacement candidates.

Of the remaining materials (In, Sn and Ag), indium and tin coatings are closest in terms of frictional behaviour to lead, with silver yielding higher friction at or around room temperature [2]. On this basis it would appear that the strongest candidates are indium and tin. Of these, indium is chemically the less reactive and was therefore selected as the prime candidate.

Furthermore, manufacture of indium-bronze cages, containing free pockets of indium (like those of lead in lead-bronze cages) should be feasible, as the formation of free indium pockets should occur with the addition of >11% indium to the bronze (based on the solubility of indium in copper).

2. PHYSICAL VAPOUR DEPOSITION (PVD) OF INDIUM

Indium coatings were produced by the process of ion-sputter deposition; the same process used at ESTL to produce lead and MoS_2 coatings. A coating procedure very similar to that used for lead coatings was adopted for indium on the basis that the two metals have broadly similar physical properties. However, the melting point of indium (157°C) is approximately half that of lead (328°C); this meant that a lower sputter target current was used to prevent the target from melting.

3. PRODUCTION OF COATINGS ON DISCS

Initial attempts were made to create a thin film of indium on 52100 steel test discs. This approach was taken for two reasons. Firstly, the discs are of a size convenient for subsequent analysis and thickness measurement of the film, and secondly, the manner in which they are coated is representative of the way that certain tribological components (e.g. gears and bearing raceways) would be coated.

A target coating thickness of 0.1μm was chosen - for two reasons:

a) As this order of thickness would be required for subsequent SOT testing.
b) The morphology of PVD lead coatings undergoes a change at a thickness of ~0.1μm, (Figs. 2 & 3).

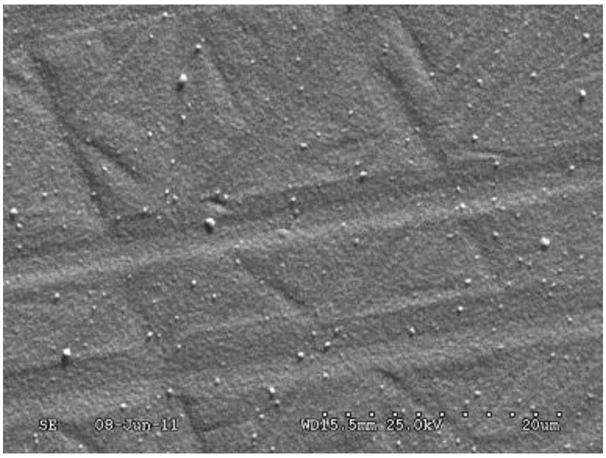

Figure 2. SEM image of 0.1μm lead thin-film on 52100 steel disc.

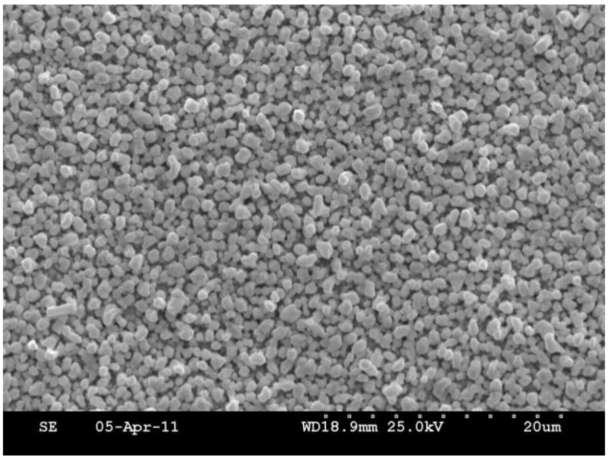

Figure 3. SEM image of 1µm lead thin-film on 52100 steel disc.

For lead coatings of thickness ≤0.1µm the coating is mostly smooth with a few small globules of lead beginning to make an appearance. For thicker coatings (e.g. 1µm, Fig. 3) the surface of the coating has an entirely globular structure. It was of interest to know whether indium coatings displayed a similar behaviour.

4. ANALYSIS OF DISC COATINGS

4.1. Coating Thickness and Adhesion

Coating thickness was measured using a Fischerscope XDL 419 X-ray fluorescence (XRF) spectrometer (calibrated using a Fischer indium film standard of thickness 0.68µm).

An initial indium coating was produced to determine the deposition rate of indium. Knowledge of the deposition rate enabled a second coating run to be made to achieve the required 0.1 µm thickness (Tab. 2).

Table 2. Details of indium disc coatings.

Coating Run No.	Coating Thickness (µm)	Standard Deviation (µm)	Adhesion Test
1	0.24	0.010	No removal of coating.
2	0.10	0.006	No removal of coating.

The standard deviation represents the uniformity of the coating. The values obtained here suggest high uniformity and are in line with those typically expected for lead coatings in the same thickness range.

Coating adhesion was checked using 19mm wide Scotch Tape (type Magic Tape 810, manufactured by 3M). The adhesive tape was pressed firmly onto the coated surface and then pulled off with a single, sharp tug. The tape was then visually examined for traces of adherent indium. This is the standard test used at ESTL for checking the adhesion of ion-sputter deposited lead films to tribological components. As shown in Tab. 2, both coatings passed the adhesion test.

4.2. Coating Morphology and Purity

Coating morphology was examined by SEM (scanning electron microscopy). All indium coatings displayed the same microstructure as typified by Fig. 4.

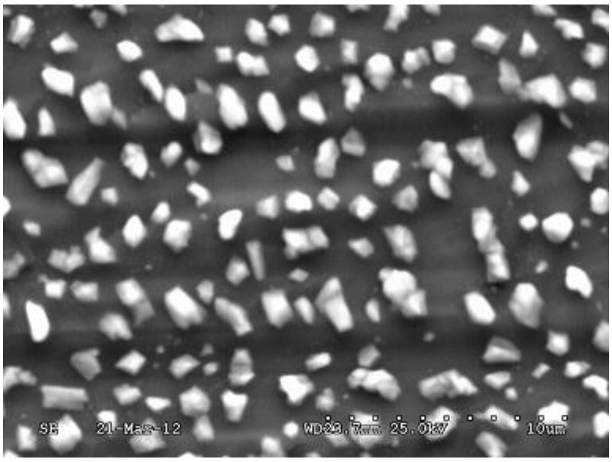

Figure 4. SEM image of 0.10µm indium thin-film on 52100 steel disc.

The indium coating consisted of isolated nodules of indium. These nodules were in the size range 1-2µm.

The chemical purity was analysed by EDAX (energy dispersive X-ray spectroscopy). A high level of purity is important in order to maintain the low shear, lubricating properties of the film. An EDAX spectrum for the 0.10µm-thick coating is shown in Fig. 5.

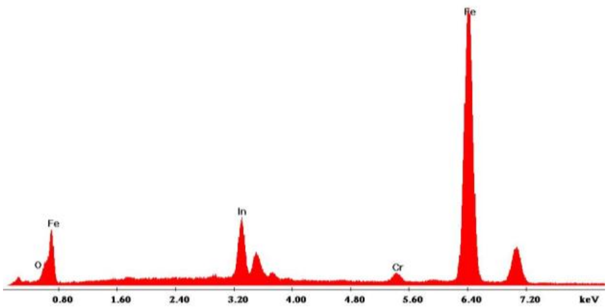

Figure 5. EDAX spectrum of *0.10µm indium thin-film on 52100 steel disc.*

The spectrum indicates a high purity. The central cluster of peaks are all for indium. The remaining large peaks are for elements in the steel substrate (iron and chromium). Oxygen has been labelled so as to indicate its position in the spectrum, but there were no visible peaks for oxygen in any of the spectra taken. This indicates that the films are, within the resolution of the instrument, essentially oxygen-free.

It should be noted that EDAX probes to a depth of 1-

2µm such that, for thin films on steel the spectra will indicate the presence of elements of the underlying steel (e.g. iron, chromium and carbon).

A spectrum obtained from an area lying between the nodules showed a small indium peak indicating that there is an underlying, albeit very thin, indium layer present.

5. PRODUCTION OF COATINGS ON BALLS

The purpose of coating balls was to enable SOT (spiral orbit tribometer) testing of the indium coating in order to assess its tribological properties.

In order to allow comparison of film performance with existing SOT data for lead and MoS_2, ball coatings of thickness in the range 0.08-0.09µm were required.

6. ANALYSIS OF BALL COATINGS

6.1. Coating Thickness and Adhesion

Coating thickness was measured and adhesion tested in the same manner as for the discs. The results are given in Tab. 3.

Table 3. Details of indium ball coatings.

Coating Run No.	Coating Thickness (µm)	Standard Deviation (µm)	Adhesion
1	0.04	0.010	Some removal of coating.
2	0.08	0.020	Some removal of coating.
3	0.06	0.016	Some removal of coating.
4	0.09	0.012	Some removal of coating.
5	0.11[a]	0.011	Some removal of coating.

[a] 0V substrate bias.

A range of thicknesses were obtained, some of which were below the target thickness. The adhesion of indium to the balls was poor compared to the discs. This could be due to the lower intensity of ion-etching (the initial 'cleaning' phase prior to coating deposition) experienced by balls compared to discs due to rig design. This was compensated however by using a longer etch time. Another possibility is that due to the structure of the coating, the rolling motion of the balls during coating may breakdown the nodules resulting in loose indium debris on the ball surface.

Coating run 5 was carried out without biasing the substrate (which has the effect of lightly etching the substrate) during deposition in order to investigate whether this would affect/improve the adhesion. However, the adhesion test result was the same.

Coating runs 2 and 4 afforded coatings of the required thickness (for the SOT tests). The adhesion was deemed sufficient for initial SOT tests to be carried out, using balls from the fourth, lower standard-deviation run.

6.2. Coating Morphology and Purity

The coating morphology on the balls was somewhat different to that of the disc coatings, see Figs. 6 and 7.

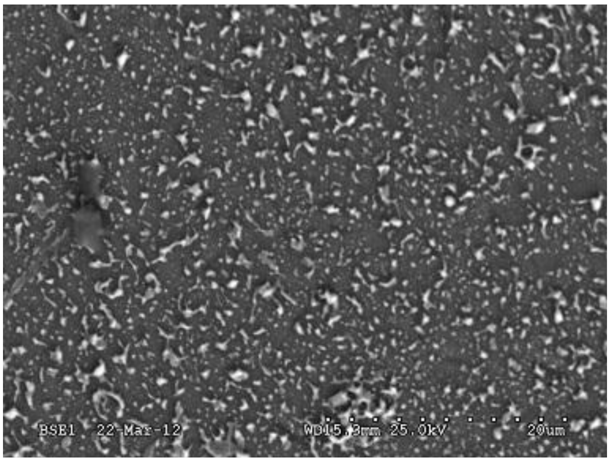

Figure 6. SEM image of 0.08µm indium thin-film on 52100 steel ball.

The coatings, again, consisted of isolated islands of indium rather than a continuous layer. However, there was a much wider variation in the particle size; probably a result of the rolling motion of the balls as they are coated.

Fig. 7 appears to show that not biasing the substrate resulted in greater coalescence of the indium particles.

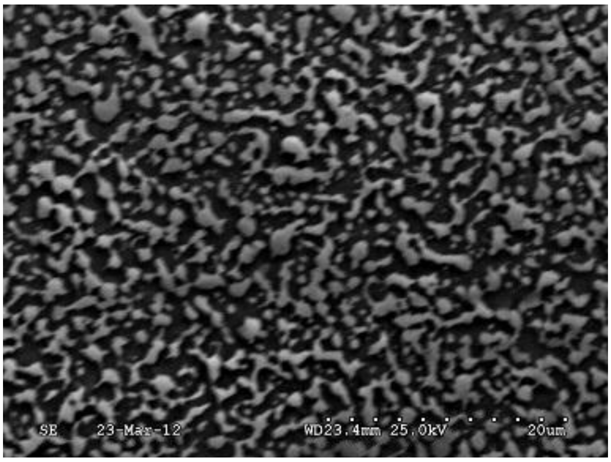

Figure 7. SEM image of 0.11µm indium thin-film on 52100 steel ball. Coating was produced without biasing the substrate.

EDAX analysis of the indium films on balls revealed the same chemical composition as for films on discs.

7. SPIRAL ORBIT TRIBOMETRY (SOT) TESTS

7.1. Apparatus

The spiral orbit tribometer is essentially a thrust bearing, with an individual ball held between two interchangeable flat plates, located within a vacuum chamber. A load is applied to the top plate via a spring-loaded linear translator. The lower plate rotates via a motor located outside the chamber, causing the ball to move in a spiral path with a radius ~21mm.

This configuration causes the ball to spiral outwards. A fixed guide plate is positioned to keep the ball within the flat plates and to produce a repeatable orbit. A force transducer behind the guide plate measures the force exerted by the ball onto the guide plate. From this, a friction coefficient value is found, once per orbit. The SOT is controlled using a LabVIEW-based data acquisition program.

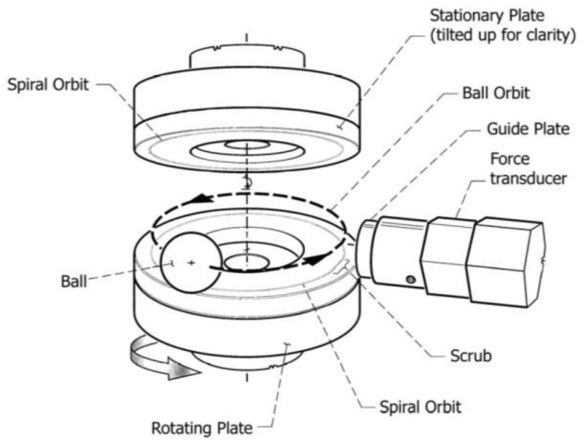

Figure 8. Internal arrangement of SOT.

The arrangement of the SOT allows the ball to experience rolling, sliding and pivoting – all motions experienced by a ball in an angular contact bearing. This provides a more representative testing regime of a lubricant than conventional pin-on-disc testing, which only recreates sliding motion. Furthermore, thin, soft-metal films (e.g. lead) typically display very poor lifetimes when assessed through sliding methods, making pin-on-disc tribometry unsuitable for this work.

7.2. Test Samples

SOT flat samples (plates) were manufactured from non-passivated 440C steel, polished to a roughness of Ra<0.05µm. Prior to testing, all plates were cleaned ultrasonically using Lenium ES solvent in accordance with standard ESTL procedure.

7.3. Test Details

Test conditions (see Tab. 4) were selected to provide meaningful comparison with previous data generated on solid lubricant coatings [5]. Three life-tests were performed, two with a coating thickness of ~0.09µm indium on the ball, and a third with a thicker coating of 0.11µm (0V substrate bias).

Table 4. Test parameters for SOT tests.

Condition	Value
Environment	High vacuum (<1.3 x 10^{-6} mbar)
Temperature	Room temperature (~22°C)
Peak contact stress	3.00 GPa
Load	~93N
Rotation speed	100 RPM
Ball diameter	7.14 mm
Ball material	52100 steel
Failure Criteria	$\mu \geq 0.3$ for three consecutive orbits

7.4. Life-Test Results

SOT life test results are summarised in Tab. 5, together with data obtained under similar test conditions for lead and MoS_2 coatings of similar thicknesses. Where multiple tests are shown, the values given are means.

Table 5. SOT lifetimes for solid lubricants (3.00GPa peak contact stress).

Coating	No. of Tests	Coating Thickness (µm)	Lifetime (orbits)	CoF at Mid-Test
Indium	2	0.09	279,888	0.022
	1	0.11	433,119	0.023
MoS_2	3	0.08	37,109	0.016
Lead	3	0.09	1,881,587	0.045

From the above it is clear that the lifetime of indium is longer than that of MoS_2 but shorter than that of lead. In addition, it shows that the life can be extended through the application of a thicker film of indium to the ball.

Indium's frictional properties are good, the friction coefficient being only slightly higher than that of MoS_2, and about half that of lead.

7.5. Post-Test Inspection

Post-test, the balls and flat plates were examined via SEM and EDAX. This analysis showed clear evidence of indium transfer from the ball to the flat plates through the action of rolling (Figs. 9 & 10). Similar observations have previously been made for lead [1].

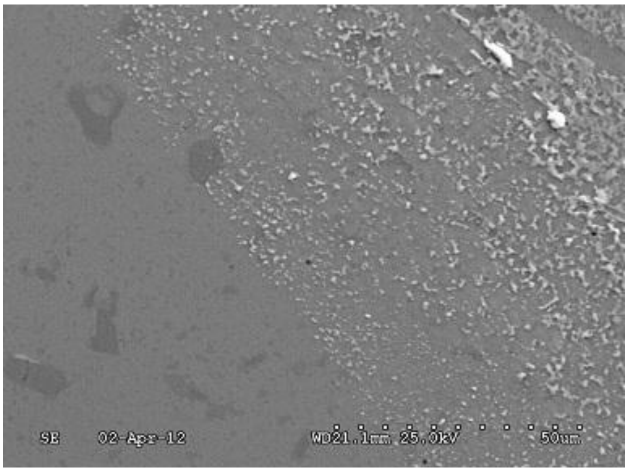

Figure 9. Edge of running track on SOT plate, showing a layer of transferred indium.

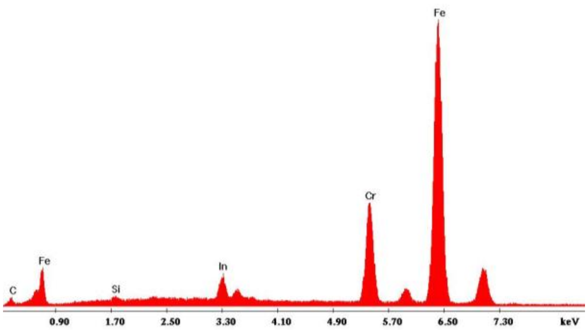

Figure 10. EDAX spectrum of running track on SOT plate, with clear indium signal. No indium peak was observed in a 'control' scan outside the running track.

8. CONCLUSIONS

The PVD indium films produced at ESTL in this initial study offer very low in-vacuum friction (μ=0.022), lower than for lead (μ=0.045) and close to that of MoS_2 when tested under the same conditions.

The in-vacuum lifetime of the current indium coatings (~280,000 orbits) is significantly higher than for MoS_2 (~40,000) but significantly lower than for lead (~2 million orbits).

The adhesion of the present indium coatings to bearing steel appears inferior to that of lead. Further optimisation of the sputtering process may improve adhesion and lead to longer lives.

9. REFERENCES

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