

Yoshinori Takeichi – Marian Dzimko – Uwe Winkelmann *

THE EFFECTS OF MoO₃ POWDER LUBRICATION ON TRIBOLOGICAL PROPERTIES OF SLIDING PAIR EXPOSED TO HIGH TEMPERATURE

The effect of molybdenum trioxide MoO₃ powder on the tribological properties of sliding pair exposed to the temperature up to 700 °C was examined. The experiment showed that MoO₃ powder placed directly on the sliding surface of main tribological body influenced and reduced friction values of tested materials. Especially in a temperature range from 500 to 700 °C a clear minimum of friction coefficient around 600 °C was observed. The X-ray diffraction analysis of friction and wear tracks that appeared on specimens showed mixture of different elements which are the product generated during tribological processes at high temperature.

Keywords: aluminium bronze, molybdenum trioxide, high temperature, Cu₃Mo₂O₉, solid lubricant

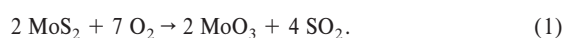
1. Introduction

In this experimental work the tribological properties of solid lubricant coated stainless steel in contact with aluminium bronze under high temperature condition were studied. It is one of the capable materials which can be used at high temperature because of their thermal stability up to around 300 °C. However, at the temperature of over 400 °C, high friction coefficient and poor wear resistance are noted that why their lubricity properties are required to be further improved. On the other hand, the lubricity of many kinds of metal oxide at high temperature was studied. It was reported that MoO₃ showed superior lubricity at high temperature of about 700 °C [1], therefore it was expected that MoO₃ layer placed on the active friction surface enhances friction properties of aluminium bronze at high temperature.

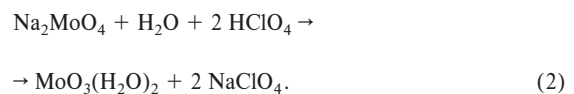
2. Material and Sample Description

2.1 Molybdenum Trioxide MoO₃

Molybdenum trioxide MoO₃ is chemical compound. This compound is produced on the largest scale of any molybdenum compound. It occurs as the rare mineral molybdite. Its chief application is as an oxidation catalyst and as raw material for production of molybdenum metal. The oxidation state of molybdenum in this compound is +6 [2]. MoO₃ is produced industrially by burning molybdenum disulfide, the chief ore of molybdenum:



The laboratory synthesis entails the acidification of aqueous solutions of sodium molybdate with perchloric acid [3]



The dihydrate loses water readily to give the monohydrate. Both are bright yellow in colour. Molybdenum trioxide dissolves slightly in water to give “molybdic acid.” In base, it dissolves to afford the molybdate anion [2]. In the gas phase, three oxygen atoms are double bonded to the central molybdenum atom. In the solid state, anhydrous MoO₃ is composed of layers of distorted MoO₆ octahedra in an orthorhombic crystal. The octahedra share edges and form chains which are cross-linked by oxygen atoms to form layers. The octahedra have one short molybdenum-oxygen bond to a non-bridging oxygen [4]. Molybdenum trioxide has widespread industrial use. Its major use is as an additive to steel and other corrosion-resistant alloys. It is also used in the production of molybdenum products, as an industrial catalyst, a pigment, a crop nutrient, a component of glass, ceramics and enamels, a flame retardant for polyester and polyvinyl chloride resins, and as a chemical reagent. [5] Because of MoO₃ layered structure (see Fig. 1) MoO₃ is of interest for intensive tribological experimental investigations conducted at elevated and high temperatures. Physical and chemical properties are listed in Table 1.

Fig. 2 shows SEM image of MoO₃ powder used in experimental investigation of friction properties for the tribological pair composed of coated stainless steel specimen rubbed against aluminium bronze.

* Yoshinori Takeichi¹, Marian Dzimko², Uwe Winkelmann³

¹ Department of Mechanical Engineering, Toyohashi University of Technology, Toyohashi Aichi, Japan, E-mail: takeichi@mech.tut.ac.jp

² Faculty of Mechanical Engineering, Department of Design and Machine Elements, University of Zilina, Slovakia

³ Institute for Mechanical Engineering (IfM), University of Applied Sciences Magdeburg-Stendal, Magdeburg, Germany

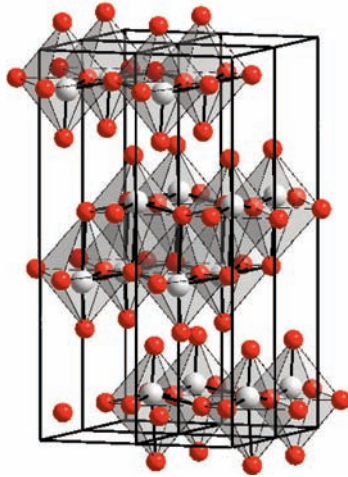


Fig. 1 Crystal structure of MoO₃ [2, 5]

Physical and chemical properties

Table 1

Appearance	White or slightly yellow to slightly bluish powder
Odour	Odourless
Solubility	0.107 g/100 g water @ 18 °C
Density	4.69 g/cm ³ , solid
Boiling Point	1150 °C
Melting Point	795 °C begins to sublime at 700 °C
Crystal structure	Orthorhombic

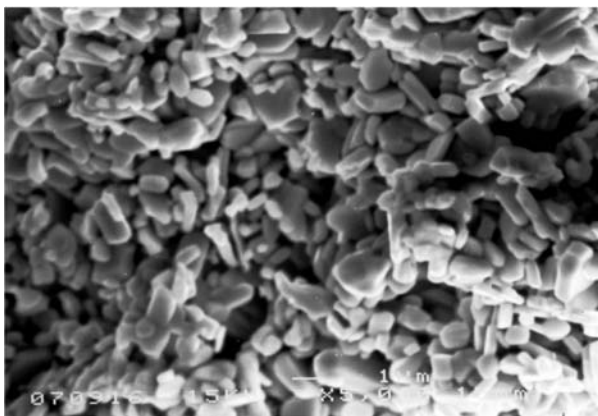


Fig. 2 SEM image of MoO₃ powder

2.2 Aluminium Bronze

Aluminium bronze, C6191 (JIS H 3250, Japanese Industrial Standards), with excellent fatigue resistance, corrosion resistance and wear resistance was chosen as material under test. The compositions of C6191 are shown in Table 2. It is copper based alloy which includes about 10 % aluminium and 3 to 5 % iron.

Compositions of aluminium bronze C6191

Table 2

Content	Cu	Al	Fe	Mn	Ni
Dens. %	81-88	8.5-11	3-5	0.5-2	0.5-2

2.3 Stainless Steel

Stainless steel, SUS304 (JIS G 4303) was used as a counterpart material for the friction test.

3. Specimens

It is well known that the oxide powders are difficult to adhere to metal surface. In the case of executed experiments the sample preparation was done as follows.

1. The sliding nominal surface of specimen was polished and then slightly sandblasted. The surface roughness (*Ra*) was measured with values around 1.0 μm.
2. The specimen was put into a glass container filled with acetone in which a defined amount of MoO₃ powder was mixed well. To achieve stable dispersion of MoO₃ powder in acetone, the ultrasonic vibration for 3 minutes was adapted. The container with the specimen was warmed up to 45 °C to evaporate acetone.



Fig. 3 Sample preparation procedure - coating

By this method finally the MoO₃ powder was uniformly accumulated on the sliding surface of the stainless steel specimen. In this way prepared specimens were used as a MoO₃ coated specimens. See Figs. 3 and 4.

3. The amount of accumulated MoO₃ powder on the specimen nominal surface was estimated by measuring the weight of the specimen before and after coating treatment. An average mass of adherent powder was controlled to be between 5 and 6 mg.

Stainless steel was used as a ring specimen and some of them were coated with the MoO₃ powder. The outer and inner diameter of the ring specimen was 20 and 15 mm, respectively.

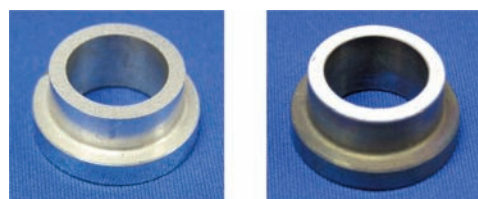


Fig. 4 Stainless steel ring specimen sandblasted uncoated and coated

Aluminium bronze was used as a disk specimen and its thickness was about 5 mm. Fig. 5 shows the form of an alloy rod and the prepared disc.

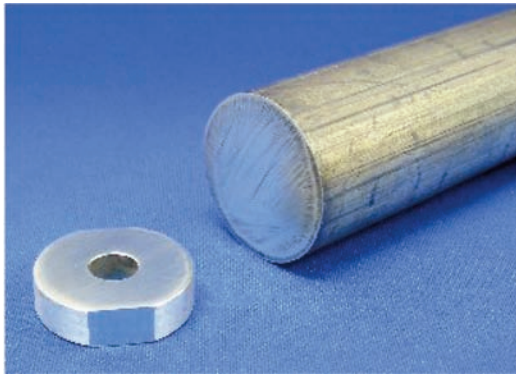


Fig. 5 Aluminium bronze disc specimen uncoated

4. Friction Test

To perform the friction test the ring-on-disk tribometer with a furnace in which the ring and disk specimens were mounted was used. The common load for all the experiment was set at 61.8 N which evocated to the median contact pressure of 0.46 MPa at the nominal surface of ring and disc. The ring specimen rotated at 60 rpm so the speed corresponded to the sliding speed of 55 mm/s. During performed experiments the approximate sliding distance reached about 200 m. For each temperature range starting at the room temperature (R.T.) and increasing step by step up to 700 °C new specimens were used. In this way the initial conditions for testing the high temperature lubrication ability of the coating remained the same for the whole experimental work. The tribometer was thoroughly described in [6, 7]. Figure 6 shows the plotted friction coefficient for the uncoated and coated specimens. It can be seen that the friction coefficient in the case of uncoated pair steadily increased from values around 0.25 to values of 0.72 cor-

responding to the increase of temperature from R.T. to 600 °C. By reaching the temperature of 700 °C the test was terminated because friction force exceeded the upper limit of this tribometer. The trend of the friction coefficient curve for the pair represented by MoO₃ coated stainless steel specimen and aluminium bronze can be divided into two different parts. The first part is characterised by a steady increase of values of the friction coefficient starting from 0.19 to 0.56 well corresponding to the increase of the temperature from R.T. to 400 °C. The second part is characterised trough a decrease of the coefficient of friction to the level of 0.44 with increase of the temperature up to 600 °C, followed by an increase of the friction coefficient again to the value of 0.57 by increasing temperature to 700 °C. This increase of the coefficient of friction around the 700 °C can be partly seen as the influence of beginning MoO₃ sublimation.

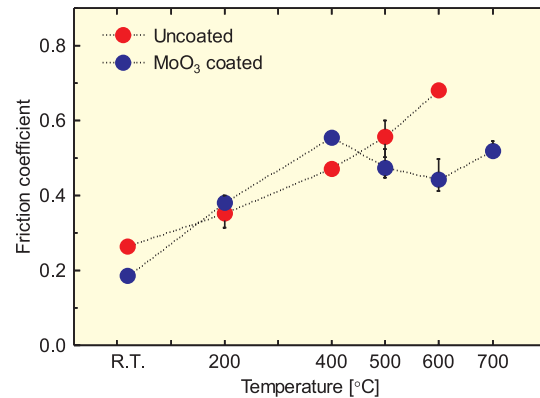


Fig. 6 Friction coefficient for the aluminium bronze disk and coated and uncoated MoO₃ stainless steel ring

From Figs. 7 and 8 the difference of adhesion appearing can be clearly seen. While for uncoated surfaces of both specimens a large amount of adhesion was observed (started at temperatures around 400 °C) for the MoO₃ coated stainless steel ring a large adhesion was observed only at temperature of 400 °C.

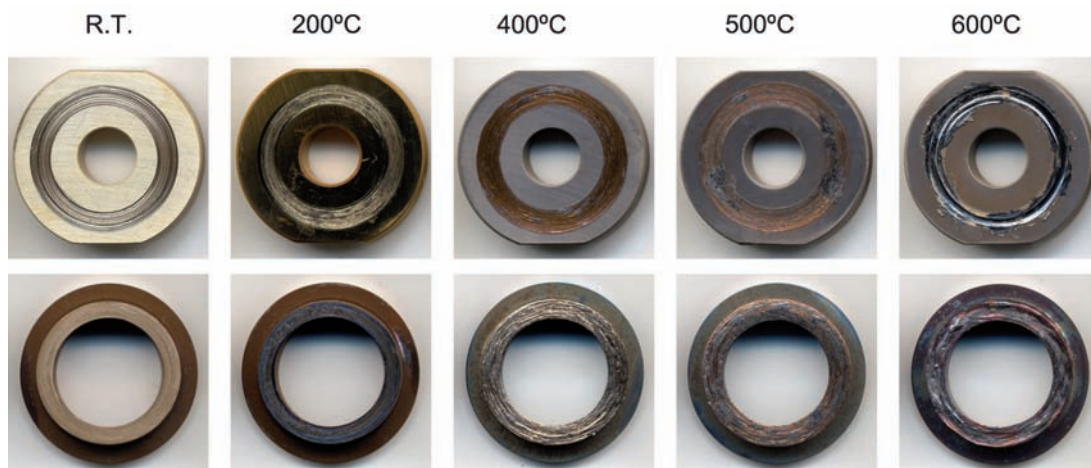


Fig. 7 Uncoated sliding surfaces of stainless steel ring and aluminium bronze disk after experiments at different temperatures

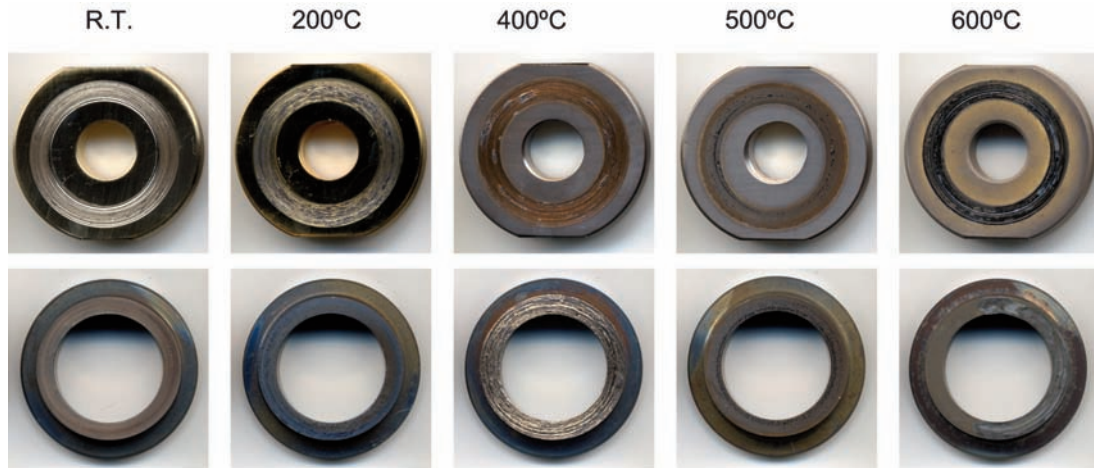


Fig. 8 MoO₃ powder coated sliding surfaces of stainless steel ring and aluminium bronze disk after experiments at different temperatures

5. X-ray Diffraction Analysis

For better understanding of friction and lubrication processes realised inside the contacted areas the XRD (X-ray diffraction) analysis was used. Some of the obtained results of the friction track on the aluminium bronze disk specimen, pressed and rubbed against the stainless steel ring coated with MoO₃, are presented in Fig. 9.

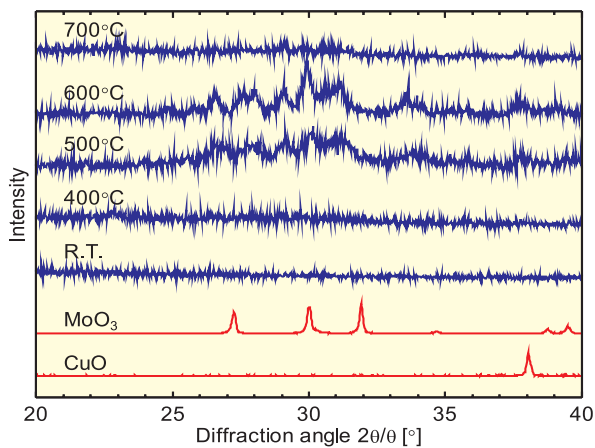


Fig. 9 XRD spectra obtained from the friction track resulting from temperatures R.T. to 700 °C of the aluminium bronze rubbed against MoO₃ coated stainless steel ring, for MoO₃ powder and for CuO powder

The diffraction peaks obtained from MoO₃ powder and CuO powder are also shown in the same figure as reference. To obtain significant data cobalt target X-ray source was chosen. This was necessary because aluminium bronze contains a few % irons as indicated in Table 2. In this range of diffraction angle, no peak of aluminium bronze could be registered. The analysis of specimen used and tested at higher temperatures of 500 and 600 °C shows several peaks with certain intensity. Fig. 9 shows that similar peaks are obtained also from the specimens tested at 700 °C,

though the diffraction intensity is small. This comparison with the diffraction peaks obtained from MoO₃ and CuO powder leads to the conclusion that the peaks obtained from the friction track on the aluminium bronze are not resulting directly from MoO₃ or CuO powders.

In our previous work [6, 7] we conducted peak identification with the diffraction peak intensity in the database. The XRD spectrum obtained from the friction track of aluminium bronze slid against the MoO₃ coated ring at 600 °C and the diffraction peak intensity of Cu₃Mo₂O₉ in the database (JCPDS 01-070-1495) were compared. It can be suggested that the emerged material on the friction track of aluminium bronze is Cu₃Mo₂O₉ because the diffraction peak position and intensity of database are in good agreement with the obtained XRD spectrum. This can indicate selective transfer of interacting elements during tribological process at high temperatures. Also it can be considered that MoO₃ reduced friction of the tested pair by changing into Cu₃Mo₂O₉. Wahl et al. reported [8] that the ion-beam deposited amorphous Cu-Mo coating on alumina substrates showed low friction coefficient at 530 and 650 °C. They suggested that the amorphous Cu-Mo coating changed to crystalline oxide CuMoO₄ and that the softened oxides worked as high temperature lubricant. Though Cu₃Mo₂O₉ has different crystal structure from CuMoO₄, it is possible that Cu₃Mo₂O₉ having similar ingredient with CuMoO₄ is softened and shows superior lubricity at high temperature.

6. Conclusions

Molybdenum trioxide MoO₃ powder in the form of an adhered layer supplied to the sliding interface between aluminium bronze and stainless steel reduced the friction coefficient of both materials at high temperature. The effect of abrasion and adhesion shifted to the temperature range of 500 and 600 °C. Copper-molybdenum oxide, which was supposed to be Cu₃Mo₂O₉, was generated during the tribological contact process by sliding aluminium bronze and stainless steel in presence of MoO₃ powder.

Acknowledgement

This work was carried out under the support of The Ministry of Education, Culture, Sports, Science and Technology (MEXT), Japan. Grant-in-Aid for Young Scientists (B): 21760112 and by VEGA project V08-036-00 of the Ministry of Education, Slovak Republic.

Exchanges were realised with the support of the National scholarship scheme of the Ministry of Education and Slovak Academic Information Agency SAIA, s.r.o. Slovak Republic.

The authors wish to thank Mrs. Naoki Okamoto, and Mr. Takashi Chujo, Masao Uemura, Ivan Havetta, Hiroto Kobayashi for their support during the intensive experimental work.

References

- [1] PETERSON, M. B., MURRAY, S. F., FLOREK, J. J.: *Consideration of Lubricants for Temperatures above 1000 °F*, ASLE Trans., 2/1960, 225-234.
- [2] http://en.wikipedia.org/wiki/Molybdenum_trioxide (02.02.2010)
- [3] HEYNES, J. B. B.; CRUYWAGEN, J. J.: *Yellow Molybdenum(VI) Oxide Dihydrate*, Inorganic Syntheses, 1986, vol. 24, pp. 191. ISBN 0-471-83441-6
- [4] WELLS, A.F.: *Structural Inorganic Chemistry*, Oxford: Clarendon Press, ISBN 0-19-855370-6, (1984)
- [5] <http://www.chinatungsten.com/Molybdenum/Molybdenum-Trioxide.html> (02.20.2010)
- [6] PETERSON, M. B., MURRAY, S. F., FLOREK, J. J.: *Consideration of Lubricants for Temperatures above 1000 °F*, ASLE Trans., 2/1960 225-234.
- [7] TAKEICHI, Y., CHUJYO, T., OKAMOTO, N., HAVETTA, I., DZIMKO, M., UEMURA, M.: *Wear and Frictional Properties of Aluminium Bronze Alloy Lubricated with Molybdenum Trioxide at High Temperature*, Synopsis of 17th International Colloquium Tribology in Esslingen 2010, ISBN-Nr. 3-924813-80-9
- [8] TAKEICHI, Y., CHUJYO, T., OKAMOTO, N., UEMURA, M.: *Effects of Molybdenum Trioxide on the Tribological Properties of Aluminium Bronze under High Temperature Conditions*, Japanese Society of Tribologists, Japan Tribology Online, vol. 4, 5/2009, pp. 135-139
- [9] WAHL, K. J., SEITZMAN, L. E., BOLSTER, R. N., SINGER, I. L., PETERSON, M. B.: *Ion-Beam Deposited Cu-Mo Coatings as High Temperature Solid Lubricants*, Surface and Coating Technology, 89/1997, 245-251.