Molybdenum based coatings on 100Cr6 bearing steel surfaces

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Abstract

Energy losses and friction locking are decisive factors in the conceptual design and sustainable realization of machine elements, as in today's industry wear and friction annually cause enormous amounts of costs. According to current estimates the direct losses due to friction and wear in industrialised countries are up to 7 % of the gross national product. Thus, improvements of tribological properties of rolling bearings by molybdenum based coatings generated on bearing surfaces represents a promising approach. Those coatings are to be optimally adapted to the load case by minimizing the slip and resulting abrasive wear by rolling elements. Therefore, molybdenum-based coatings were applied by means of RF-magnetron sputtering under defined argon atmosphere at controlled and adjusted oxygen partial pressure on 100Cr6 bearing steel surfaces. The layer systems generated, featured different stoichiometries of molybdenum and oxygen. The layers were analyzed subsequently regarding morphology and chemical composition. In further investigations, those layer systems are to be investigated regarding their tribological resistance.

1 Introduction

Currently, the control and knowledge of wear and friction behaviour of machine elements in relative motion to each other is very important. To cope with the growing competition, today's industry places high demands on innovative materials. One of the main targets is the reduction of friction and wear, as these annually cause enormous costs in the manufacturing industry. Direct losses due to friction and wear are up to 7% of the gross national product in industrialized countries [1-4]. This study includes investigations of generated molybdenum based layer systems on ASSI 552100 (100Cr6) bearing surfaces through RF-magnetron sputtering [5-7]. The approach of this project is based on the development of an oxide layer system, which consists of different phases and should act self-generatively under tribological load caused by oxidation in the near-surface area. The system consists of a Ti bonding layer, sputtered on the substrate and a Mo reservoir, that should favour diffusion into the MoO_x reaction layer. For this purpose, different coating strategies were implemented which are to be presented in this work. The structure of the angular contact ball bearings and the layer system investigated are shown in Fig. 1.



Figure 1: a) systematic structure of an angular contact ball bearing b) *schematic representation of the layer system with different layer components.*

2 Experimental

2.1 Material and preparation

The molybdenum based coatings generated were deposited on running surfaces of angular contact ball bearings made of ASSI 552100. For surface analysis cylindrical samples (diameter: 14 mm; height: 5 mm) were prepared for the PVD procedure. The surfaces were polished to an average surface roughness of about $Sa = 0,2 \mu m$ with diamond suspension prior to the coating procedure while roughness did not change significantly after the coating procedure as has already been described in detail is [4].

2.2 Magnetron sputtering

RF-magnetron sputtering was realized under argon/oxygen vacuum atmosphere with different targets. At first pure molybdenum was sputtered. By increasing the mass flow of oxygen at varied deposition power, stoichiometries of molybdenum and oxygen were adjusted reactively. To distinguish between different layer components, Ti was sputtered separately as well to determine the morphology of the bonding layer system. For all procedures which were conducted under oxygen atmosphere the deposition time was 90 min, while pure Ti was sputtered for 45 min. To generate stable molybdenum oxides in the near-surface areas a MoO₃ target was used as well as alternative to conventional reactive sputter conditions. The temperature of the substrate was 180 °C at a constant target power of 150 W. To remove the surfaces from condensates and contaminations, a RF-cleaning plasma (power: 650 W at a frequency of 13.56 MHz) was used prior the coating procedure.

2.3. Surface analysis

A scanning electron microscope (Zeiss Supra 55 VP) equipped with different detectors (backscattered electron (BSE), secondary electron (SE) and Inlens detection) was used for high resolution analysis. Chemical composition of representative areas was probed using energy dispersive X-ray spectroscopy (EDX) with a Bruker Quantax XFlash ® SDDs 125 eV detector. EDX data was collected at an acceleration voltage of 10 kV. The SE, BSE and Inlens images were recorded at lower acceleration voltage (5 kV), based on the very thin molybdenum (oxide) layers.

To get knowledge about layer thickness, high resolution cross sections were cut from the layers with a focused ion beam (Zeiss Auriga). This combined characterization method was needed, assuming the sputtered layers being very thin.

3 Results

Information about the morphology and the chemical composition of the surfaces were obtained by surface sensitive analytical methods. Figure 2 a) shows secondary electron images of a sputtered Molybdenum layer that was generated reactively. The layer is completely uniform with particles that vary in size between 100 nm and 1 μ m. Figure 2b) shows a surface which was sputtered reactively. The particles are of small size of about 100 nm and do not vary in size, while oxide particles seem to have coarsened for sputtering procedures which have been realized with the MoO₃ target. The surface features small cracks which are probably caused to higher residual stresses in the layer system (cf. Fig. 2c)). The sputtered Ti layer is not completely uniform and features small areas that have not been coated. The particle size is homogen with an average diameter of about 50 nm (cf. 2d)).



Figure 2: SE images of different sputter conditions 2a) pure Molybdenum 2b) Molybdenum oxide (sputtered reactively) 2c) Molybdenum oxide (sputtered with MoO₃ target) 2d) pure Ti

For both molybdenum based oxide coatings, cross sections were prepared using focused ion beam to provide for high resolution information. Fig. 3 shows SE images of a cross section of the sputtered molybdenum based oxide systems. The layer generated from the MoO_3 target features a Mo reservoir of about 2 µm with an oxygen and molybdenum rich phase above (cf. Fig. 3 a)). The Ti bonding layer is not recognizable for both specimen, probably caused to diffusion effects. The specimen sputtered reactively features a very thin oxide layer in the contact area above a molybdenum reservoir which has a thickness of about 2.5 µm.



Figure 3: cross sections prepared using focussed ion beam 3a) Molybdenum oxide (sputtered with MoO₃ target) 3b) Molybdenum oxide (sputtered reactively)

4 Conclusion

The present work shows results of the layer generation of molybdenum-based multilayer systems on bearing steel that were applied by different sputtering strategies. Both layer systems have oxide-rich phases of different intensities in the contact area and should be tribologically tested in future investigations.

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Literatur

- [1] Czichos, H., Habig, K.H., "Tribologie-Handbuch Tribologie, Tribomaterialien, Tribotechnik", 3rd edn, Viehweg + Teubner Verlag, Wiesbaden, 2010.
- [2] Dominguez, A., Dutt, A. and De Melo, O., "Molybdenum oxide thin film for heterojunction solar cells," 44th Photovoltaic Specialist Conference, 2017, DOI: 10.1109/PVSC.2017.8366104.
- [3] Khatri, H., Collins, R. W. and Marsillac, S., "In-situ and ex-situ studies of molybdenum thin films deposited by rf and dc magnetron sputtering," 35th Photovoltaic Specialist Conference, 2010, http://dx.doi.org/10.1155/2012/723714.
- [4] Schöler, S., Wulff, D., Yilkiran, D. and Beherens, B.-A., "Inductive heat treatment as an alternative tempering method for the selective oxidation of 1.2379 tool steel surfaces," Dry Metal Forming OAJ FMT, 2015, 13-17.
- [5] Dai, X., Zhou, A., Feng, L., Wang Y., Xu J. and Li J., "Molybdenum thin films with low resistivity and superior adhesion deposited by radio-frequency magnetron sputtering at elevated temperature," Elsevier B.V., 567, 2013, 64-71, DOI: 10.1016/j.tsf.2014.07.043.
- [6] Pape, F., Dellinger, P., Moehwald K. and Poll G., "Nano- and Micro-Tribological Investigations on Molybdenum Based Coatings for Application on Bearing Surfaces," 7th International Conference on Tribology in Manufacturing Processes, 2016.
- [7] Li, W., Yan, X., Aberle, A. G. and Vankataraj S., "Adhesion Improvement and Characterization of Magnetron Sputter Deposited Bilayer Molybdenum Thin Films for Rear Contact Application in CIGS Solar Cells," International Journal of Photoenergy, 2016, Article ID 2124087, 10 pages