

## Tribological behavior of DLC-coated articulating joint implants

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### ABSTRACT

Coatings from diamond-like carbon (DLC) have been proven to be an excellent choice for wear reduction in many technical applications. However, for successful adaption to the total joint replacement field, layer performance, stability and adhesion in realistic physiological setups are quintessential and these aspects have not been consistently researched. In our team's efforts to develop long-term stable DLC implant coatings, test results gained from a simplified linear spinal simulator setup are presented. It is shown that metal-on-metal (MoM) pairs perform well up to 7 million loading cycles, after which they start to generate wear volumes in excess of 20 times those of DLC-coated implants. This is attributed to the roughening observed on unprotected metal surfaces. Furthermore, we illustrate that in contrast to DLC-on-DLC, MoM tribopairs require protein-containing media to establish low-friction conditions. Finally, results of defect monitoring during testing are presented, showing catastrophic failure of layers whose interfaces are too weak with respect to the stress-corrosion-cracking mechanism encountered *in vivo*.

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### 1. Introduction

In the field of joint implants, metal-on-metal (MoM) pairings are a viable choice because of their comparatively good machinability, leading to very smooth and conforming surfaces [1]. However, such surfaces, which are typically composed of a Co–Cr–Mo alloy, are highly susceptible to mechanical damage [2], giving rise to the demand for a more wear-resistant modification or coating. Diamond-like carbon (DLC, amorphous hydrogenated carbon a-C:H) is a candidate for coatings of biomedical implants as shown by its success in mechanical applications and its proven bioinertness [3]. DLC can be deposited on most substrate materials by a variety of plasma processes and has been the focus of intense research over the past decades (e.g. [4–7]). However, peculiarities encountered in the *in vivo* environment have thus far barred widespread implant application; examples include increased wear against polyethylene in synovial fluid [8] and unpredicted adhesion failure [9]. The unreviewed transfer of techniques learned in other fields of application to the biomedical environment seems to be especially dangerous in the case of a-C:H coatings.

For the development of successful DLC-coated implants, it is thus necessary to perform tests under conditions resembling the *in vivo* situation, as in the previous work of Lappalainen et al. [10]. In this work, these coatings are applied to spinal disk replacement implants, which represent a favored alternative to fixation

and for lumbar spinal segments are frequently ball-on-socket type [11]. However, spinal motion is biomechanically complex and not easy to simulate as it involves simultaneous bending and rotational modes under dynamic loads of up to 2 kN (cf. ISO 18192-1, [12]). In this paper, we present results attained in a simulator setup running a simplified spinal motion and discuss possible effects that lead to poor layer performance or coating failure.

### 2. Materials and methods

Ball-on-socket implant pairs made from Co28Cr6Mo (radius of curvature 14.5 mm; for further details, see Refs. [13,14]) were cleaned ultrasonically in 50/50 ethanol/acetone and introduced into the coating system (base pressure  $5 \times 10^{-6}$  Pa). The samples were cleaned *in situ* by Ar sputter cleaning from a 13.56 MHz radiofrequency Ar plasma ignited from the sample holder. Immediately after this step, 4 μm thick a-C:H layers were grown in an acetylene plasma. For comparison, a rough 16 μm DLC layer exhibiting “cauliflower” structure (“rough DLC”) was also investigated to assess the error tolerance. The resulting layers had a hardness of 23 GPa and an intrinsic stress of 3.8 GPa at a thickness of 4 μm as assessed by nanoindentation (MTS nanoindenter XP, Berkovich tip) and from the bending radius of coated silicon strips via the Stoney equation (P-10 diamond stylus profilometer, KLA Tencor), respectively. Metallic interlayers were used to provide stable layer adhesion as we describe elsewhere [15]. For studies of the system's error tolerance regarding interface stability and defects, a silicon-based interlayer commonly used to promote adhesion was

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deposited from tetramethylsilane ( $\text{Si}(\text{CH}_3)_4$ ) at a controlled oxygen contamination of  $7 \times 10^{-2}$  Pa established by a leak valve; the deposition processes' gas compositions up to  $m/q = 100$  were time-dependently recorded with a sputter process monitor (SPM200, Pfeiffer Vacuum).

Samples coated as described and uncoated reference samples were subjected to wear tests in a setup consisting of six identical testing stages. The motions applied by these stages perform a simplified version of the linear part of the human lumbar spine lateral and flexion–extension motions of  $\pm 2^\circ$  and  $+6^\circ/-3^\circ$  tilt, corresponding to wear paths of 2.0 and 4.6 mm per cycle, respectively. These motions were generated from linear actuators via a lever mechanism (Fig. 1). Wear cycles were applied at a frequency of 3 Hz under a constant perpendicular load of 1200 N applied by means of a bellows cylinder. For a change of the movement type, the samples were dismantled and remounted onto the stages in a  $\pm 90^\circ$  position after a maximum of 1 million cycles; half of the total cycle number consisted of the  $\pm 2^\circ$  and half of the  $+6^\circ/-3^\circ$  motion type. As such, these tests do not qualify as an ISO 18192-1 compliant testing, which requires three simultaneous motions under dynamic load, but are intended as a prescreening preceding further certification (for a similar setup, see Ref. [16]). Unless noted otherwise, experiments were performed in 30 g/l protein-containing bovine serum-based wear-testing fluid (Hyclone®, Cat. No. SH30856.04, Thermo-fisher) heated to  $37^\circ\text{C}$  and stabilized with anti-fouling agents ( $\text{NaN}_3$ , protease inhibitors), which served to prevent protein degradation and bacterial growth. The medium was periodically exchanged after 1 week or 1 million cycles of running time. Time-resolved data were logged at fixed intervals of 1000–5000 cycles and include actuator position, load (from a load cell positioned below the bellows cylinder, Fig. 1), and motor current; one stage was additionally equipped with strain gauges mounted at the sensor axle and used to derive an estimate of the friction coefficients, which also gives a cross-reference for the time-resolved motor current data obtained from the other stages.

Motion control, data logging and data evaluation for all stages were performed by a program running within the LabView (National Instruments) environment and additional software developed for this setup.

After a total of 0.2, 0.5, 1, 2, 5, 7, 10, 14 and 20 million cycles, samples were removed from the simulator stages and cleaned according to ISO 14242-2. The wear volume loss was derived from weight measurements (AE-163, Mettler Toledo,  $10 \mu\text{g}$  resolution) using densities of  $8.29 \text{ g cm}^{-3}$  for CoCrMo [16] and  $2.80 \text{ g cm}^{-3}$

for DLC [5], respectively. Optical micrographs and surface roughness measurements by means of a diamond stylus profilometer (P-10, KLA Tencor) gave further assessments of the implant condition; the roughness was evaluated from three  $100 \mu\text{m}$  scans taken near the implant centre, while  $2000 \mu\text{m}$  scans were taken for evaluation of the lubrication regime. Where required, more detailed investigations were performed by scanning electron microscopy (XL30 ESEM-FEG, Philips). The total number of 20 million friction cycles (i.e. 10 million of the lateral plus 10 million of the flexion–extension type) was selected as a benchmark approximately corresponding to the clinical lifetime (ISO 18192-1 annex A.5).

Additional experiments were performed to examine the lubrication conditions in the parametric surroundings of the default conditions used in this study (1200 N, 3 Hz motion frequency). Both MoM and DLC-on-DLC (DoD) pairs were subjected to wear tests at different loads, ranging from 200 to 1500 N, and motion frequencies from 1 to 5 Hz (“Stribeck surface” [17]). As well as the protein-containing wear-testing fluid (SBF), phosphate-buffered saline solution (PBS, Sigma Aldrich) and ultrapure water were additionally used as lubrication media for comparison. To exclude run-in effects, sample pairs that had run 20 million friction cycles in default conditions (1200 N load, 3 Hz motion frequency, SBF) were selected for these tests and run-in for at least 1000 cycles immediately before experiments until stable conditions were reached. Following this run-in, the friction coefficient was gathered as a function of applied load and motion speed (articulation frequency).

### 3. Results and discussion

#### 3.1. Lubrication

The Stribeck surface data allow for an analysis of the lubrication regimes present within the operational envelope of the simulators. From the Stribeck surfaces (Fig. 2), it is clear that substantial differences exist for MoM and DoD tribopairs running in water and PBS. For the lower motion frequencies and higher loads, the uncoated MoM samples exhibit a stick–slip mode of motion with very high damage to the sample surfaces. This effect is less pronounced for PBS than for water, and the coefficient of friction is much lowered, indicating a change in the electrostatic or tribochemical reactions at the CoCrMo surfaces. DoD sample pairs in contrast exhibit stable friction under these conditions and in both environments. It can

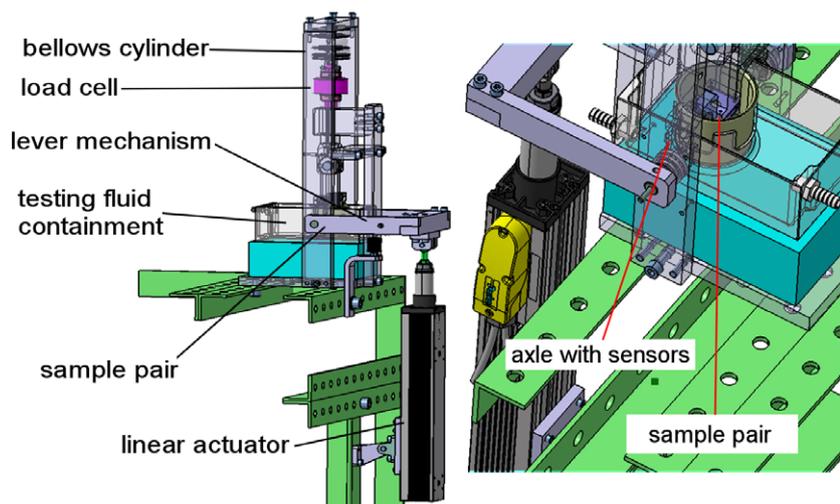


Fig. 1. Schematic of the simplified spinal simulator setup used in this study.

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