Influence of pulsed bias voltage on the tribological and morphological properties of DLC coatings deposited by an anodic arc method

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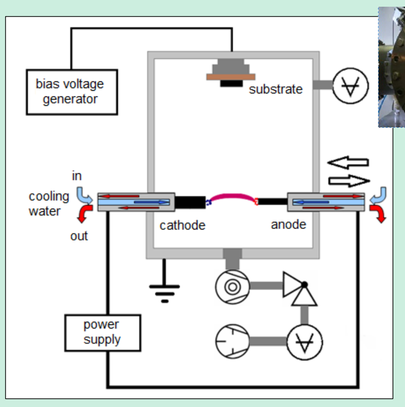
Aim:

Optimization of the tribological and morphological properties of DLC coatings to improve the biocompatibility and increase the durability of coated metal joint implants**.**

Approach:

In order to reduce friction and wear particle sizes of metal joint implants and increase their biocompatibility, thin nanostructured diamond-like carbon (DLC) coatings are applied. This is done in a PVD process, in which an arc is ignited by means of DC-discharge between two graphite electrodes. The arc is maintained for a period of 180 seconds while the distance of the evaporating graphite electrodes is kept constant. During the coating process, the substrates are exposed to a negative pulsed bias voltage that can be varied between 0 V and -1250 V. Its frequency and pulse width are also variable. The deposited layers are then analyzed with Raman-Spectroscopy to determine their morphology. The thicknesses of coatings are measured using a profilometer. Together with the difference of the weight before and after coating process, the density can be calculated and correlated with the result of Raman-Spectroscopy. Subsequently, the coated substrates are, in pairs, exposed to a fretting test in a medium of demineralized water with a tribometer. Here the coefficient of friction is investigated and correlated with the likewise conducted roughness test. The abrasion particles are made available for expertise in a scanning electron microscope by microfiltration and their size distribution is determined by means of a laser diffraction spectrometer. The aim of this investigation was to expose the influence of the bias voltage on the tribological and morphological properties of DLC coatings in particular in the range of -750 V to -1200 V bias and to find suitable values for frequency and pulse width of the bias voltage.

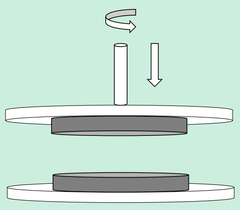
Deposition of DLC coatings by anodic arc



Coating chamber (schematic diagram)

deposition parameters	conditions
deposition pressure	5×10^{-6} mbar
arc current	80 A
arc voltage	20 V
deposition time	3 min.
ratio of diameter cathode vs. anode	2/1
pulsed bias to substrate with different frequency and pulse width	–750 V, –900 V, –1050 V, –1200 V

Wear testing setup (disc-on-disc)



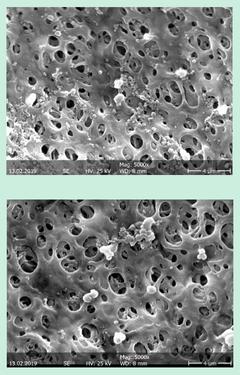
wear testing parameters	conditions
normal force	50 N
lubrication	deionized water
rotation velocity	0.025 m/s
testing time	1 h

The wear particles are generated by a Wazau TRM 1000 tribometer.

Samples

Before and after fretting test

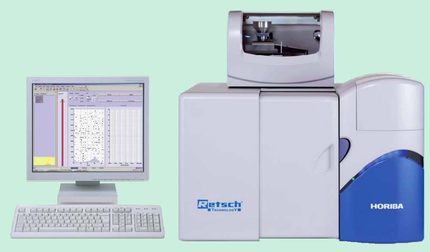
Investigation by SEM



Particles were filtered by a 0.2 µm membrane and prepared for SEM.

Particle size characterization

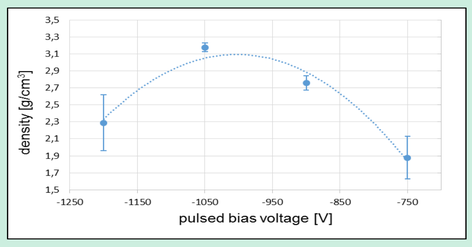
The wear particles are collected in suspension in deionized water.



The volume density distribution $q_3(x_i)$ of particle sizes were measured by Horiba LA-950 laser diffraction spectrometer.

Experiments and Results:

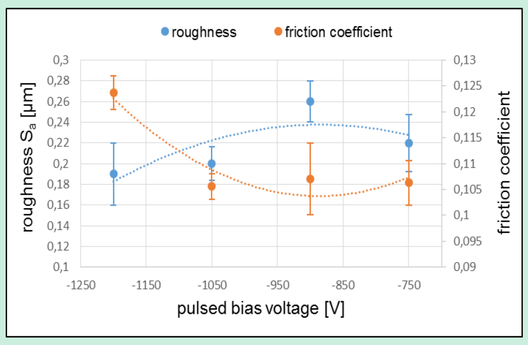
Density as a function of bias voltage



	sp^3 (%)	H (at. %)	Density (g/cm³)	Hardness (Gpa)
Diamond	100	0	3.515	100
ta-C	80-88	0	3.1	80
a-C:H hard	40	30-40	1.6-2.2	10-20
a-C:H soft	60	40-50	1.2-1.6	<10
ta-C:H	70	30	2.4	50

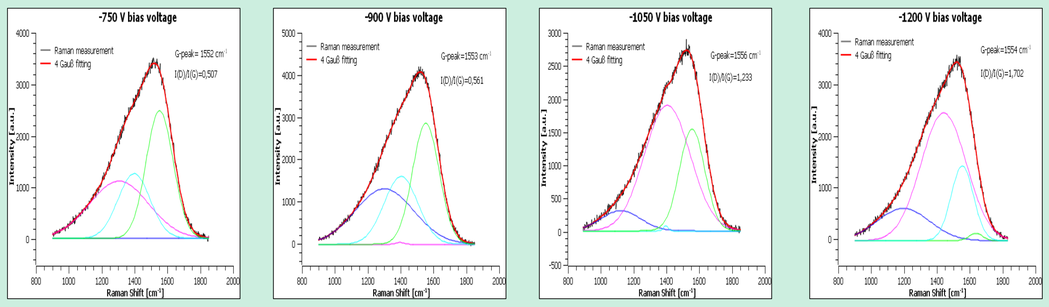
[1] J. Robertson, Materials Science and Engineering R 37(2002) 129-281.

Friction coefficient and roughness as functions of bias voltage



The roughness decrease by trend with increasing bias voltage while the friction coefficient increase.

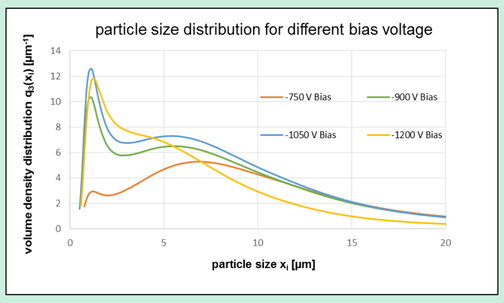
Raman spectra fitted with four-Gaussian

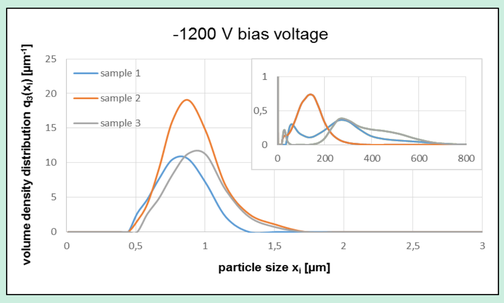


D-peak position is between 1397 cm^{-1} and 1441 cm^{-1}.
G-peak due to amorphous carbon lies between 1552 cm^{-1} and 1556 cm^{-1}.
The norminal graphitic G peak position is approximately 1588 cm^{-1}. [2]

[2] R.N. Tarrant, et al. Diamond and Related Materials 13 (2004) 1422-1426.

Volume density distribution q_3 [µm] of wear particles

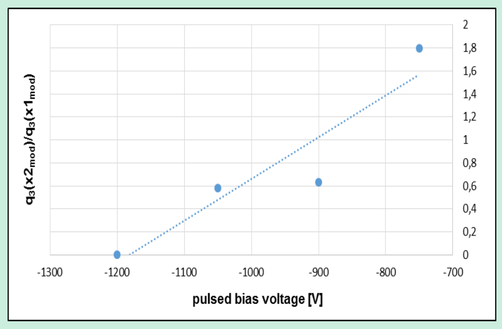




The particle size distribution shows no second maximum for a bias voltage of -1200 V.

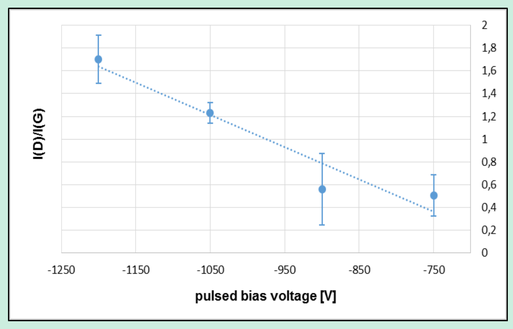
It could be shifted to a range of 1 µm by choice of suitable values for frequency and pulse width.

Ratio of $q_3(x2_{mod})/q_3(x1_{mod})$ as a function of bias voltage



With rising bias voltage the content of greater particles decrease.

I(D)/I(G) as a function of bias voltage



The ratio of I(D)/I(G) shows continuous growth with increasing bias voltage.

Conclusions:

The density of the deposited carbon layers have their maximum at -1050 V bias voltage in the range of ta-c.
The peak value of roughness lies at -900 V bias voltage. The friction coefficient tends to increase with rising bias voltage.
Raman spectra can be fitted well by Gaussian distribution based on four peaks. I(D)/I(G) increases continuously with increasing bias voltage.
At -1200 V bias, with a suitable frequency and pulse width selection, almost all wear particles are in a size range of less than 1 µm.