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Research Paper

The High performance of nanocrystalline CVD diamond coated hip joints in wear simulator test

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ABSTRACT

The superior biotribological performance of nanocrystalline diamond (NCD) coatings grown by a chemical vapor deposition (CVD) method was already shown to demonstrate high wear resistance in ball on plate experiments under physiological liquid lubrication. However, tests with a close-to-real approach were missing and this constitutes the aim of the present work. Hip joint wear simulator tests were performed with cups and heads made of silicon nitride coated with NCD of ~10 μm in thickness. Five million testing cycles (Mc) were run, which represent nearly five years of hip joint implant activity in a patient. For the wear analysis, gravimetry, profilometry, scanning electron microscopy and Raman spectroscopy techniques were used. After 0.5 Mc of wear test, truncation of the protruded regions of the NCD film happened as a result of a fine-scale abrasive wear mechanism, evolving to extensive plateau regions and highly polished surface condition (Ra < 10 nm). Such surface modification took place without any catastrophic features as cracking, grain pullouts or delamination of the coatings. A steady state volumetric wear rate of 0.02 mm^3/Mc, equivalent to a linear wear of 0.27 μm/Mc favorably compares with the best performance reported in the literature for the fourth generation alumina ceramic (0.05 mm^3/Mc). Also, squeaking, quite common phenomenon in hard-on-hard systems, was absent in the present all-NCD system.

1. Introduction

Nanocrystalline diamond (NCD) coatings grown by the chemical vapor deposition (CVD) are characterized by having diamond grain sizes typically in the range of 3–100 nm, with a relatively large fraction of hydrogen and amorphous carbon phases at the grain boundaries (Williams 2011; Liu et al., 2007). Due to the very small grain size, the intrinsic surface roughness of NCD films deposited on ultra-polished quartz or silicon substrates varies between 8 nm and 15 nm (RMS roughness) (Liu et al., 2007). These morphological and topographic characteristics, together with diamond's extreme hardness and chemical inertness, are key factors for the application of NCD coatings in tribology. In fact, several works on the tribological

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behavior of NCD-on-NCD contacts have been demonstrated the excellence of this tribopair (Amaral et al., 2010; Abreu et al., 2006, 2009; Mubarok et al., 2008; Salgueiredo et al., 2013). Following a short-time running-in period where the protruding nanodiamond asperities suffer from truncation and blunting, the NCD-on-NCD sliding system attains a steady-state friction regime with very low friction coefficients (0.01–0.05), characteristic of highly polished passivated surfaces (Abreu et al., 2006, 2009; Mubarok et al., 2008; Salgueiredo et al., 2013). A very mild wear regime is in line with this friction response, with low wear coefficient values in the range of 10^{-9} mm^3 N^{-1} m^{-1} for water lubricated contacts (Abreu et al., 2009) and one order of magnitude higher for dry systems (Abreu et al., 2006). A self-polishing micro-scale abrasive wear mechanism controls the wear behavior of the NCD-on-NCD tribosystems (Salgueiredo et al., 2013). A decisive feature for the good behavior of NCD coatings in tribology is the adhesion level rendered by the use of silicon nitride (Si_3N_4) ceramics as substrates (Almeida et al., 2007) due to the low thermal expansion coefficient mismatch and grain-to-grain epitaxial relationship between this ceramic and the diamond crystals, assisting the interface strength (Almeida et al., 2011). Additionally, Si_3N_4 materials provide not only superior mechanical properties as a structural ceramic but also excellent biocompatibility (Guedes e Silva et al., 2004; Taylor et al., 2010). High threshold loading values before coating delamination for dry systems (60 N; 3.53 GPa) (Salgueiredo et al., 2013) and for water lubricated ones (85 N; 3.96 GPa) (Abreu et al., 2009) were reported for ball-on-plate sliding experiments with NCD coated silicon nitride ceramic samples.

Having in mind the former considerations about the adequacy of NCD for tribology, summed up with NCD's biocompatibility (Amaral et al., 2008, 2009; Thalhammer et al., 2010; Bendali et al., 2014), a great potential is foreseen for the application in biotribological systems. Aiming at this focus, works were performed under the lubrication with physiological fluids. In particular, NCD-on-NCD tribopairs were tested in bovine serum media, anticipating the performance of articulating surfaces of a novel joint prosthesis. Steady state friction coefficient values of 0.10 were recorded (Amaral et al., 2008), higher than the values found for ceramic-on-ceramic and ceramic-on-metal (0.002–0.07), but considerably lower than for metal-on-metal combinations used in bearings for artificial hip joints (0.22–0.27) (Jin et al., 2006). The main reason for the friction value of the cited NCD coating lies on its relatively high starting surface roughness (RMS of 200–400 nm) (Amaral et al., 2008) when compared to 5–10 nm (Ra) for the ceramics (Jin et al., 2006). However, the wear coefficient values measured for NCD self-mated pairs in ball-on-plate tests under physiological liquid lubrication are of the order of $\sim 10^{-9}$–10^{-8} mm^3 N^{-1} m^{-1} (mild to very-mild wear regime), values that are equal or even better than the wear factors assessed in similar test conditions for metal-on-metal ($\sim 10^{-7}$ mm^3 N^{-1} m^{-1}), UHMWPE-on-metal ($\sim 10^{-7}$ mm^3 N^{-1} m^{-1}) and ceramic-on-ceramic ($\sim 10^{-8}$ mm^3 N^{-1} m^{-1}) contacts (Bendali et al., 2014).

Nowadays, there is a real concern in developing solutions for long-term performance of total hip replacement (THR) joints. Life expectancy is increasing and also is the number of young patients who present hip damage due to severe sport injuries or accidents. This is a great challenge for materials like NCD with superior biotribological behavior. The present work discusses the results of 5 Mc of test in a hip wear simulator apparatus of all-NCD coated Si_3N_4 ceramic hip joints. Si_3N_4 ceramic substrates are produced by conventional powder technology route followed by machining with diamond hard tools into femoral head and acetabular cup geometries. The NCD coatings were grown on the contacting regions of Si_3N_4 components by the hot filament CVD process.

2. Experimental

Femoral heads and acetabular liners, both of 28 mm nominal diameter, made of silicon nitride (Si_3N_4) bulk material, were fabricated according to the powder technology process described elsewhere in detail (Rodrigues et al., 2013). In brief, silicon nitride powder (AMPERPRESS Grade P, H.C. Starck) was uniaxial pressed at 160 MPa into cylindrical shape (40 mm diameter and 33 mm height) and further machined into adequate dimensions, assuming 1.225 of shrinking green-sintered constant. The compacts were pressureless sintered in a conventional graphite furnace at 1750 °C/3 h in a nitrogen atmosphere. Surface finishing was firstly done by diamond tool machining to adjust to final dimensions and then by manual polishing steps, using different grit sizes.

Concerning the geometrical data of the head and the acetabular liner, these were as shown in the Table 1. The measurements were done in a tridimensional Coordinate Measuring Machine (Mytutoyo Legex 9106 model), with a scanning ball probe of 4 mm diameter.

A NCD coating of about 10 μm in thickness was grown on the Si_3N_4 surface by the hot filament CVD method, under the following deposition conditions: current intensity of 66 A; CH_4/H_2 flow ratio of 0.02; total pressure of 25 mbar; filament temperature of 2250 °C; substrate temperature of 650 °C; gas flow of 100 ml/min; deposition time of 12 h. Prior to deposition, the Si_3N_4 ceramic surface was abraded for 1 h in ultrasonic bath with 6 nm sized diamond powder in ethanol suspension. The surface finishing of the specimens was performed manually by using soft cloths with diamond suspensions in a sequence of micrometrical particle sizes from 9 μm to 3 μm and finally colloidal silica.

Table 1 – Geometrical data of the NCD coated silicon nitride ceramic head and acetabular liner obtained from CMM measurements.

Specimen	Diameter [mm]	Radial clearance [μm]	Sphericity deviation [μm]
Head	27.88	78	14.1
Liner	27.96		3.4