



# Extremely low wear rates in hip joint bearings coated with nanocrystalline diamond

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## ARTICLE INFO

### Article history:

Received 24 June 2014

Received in revised form

18 August 2014

Accepted 7 December 2014

Available online 17 December 2014

### Keywords:

Nanocrystalline diamond

Wear simulation

Ceramic hip joint

## ABSTRACT

A hip joint wear simulator is used for the first time to evaluate the performance of acetabular liners and femoral heads made of silicon nitride ceramic coated with nanocrystalline diamond (NCD), grown by a hot filament chemical vapor deposition (HFCVD) method. Wear is assessed by gravimetry, by which volume and linear wear are estimated. Even with only one million cycles of test, a very stimulating finding is the extremely low wear rate of the head after the diamond polishing running-in step, of about 0.005 mm<sup>3</sup>/million cycles (Mc). This corresponds to a linear wear of 0.08 μm/Mc, one order of magnitude better than the best value currently known for ceramic-on-ceramic hip joints. Small scale abrasion is found as the dominant wear mechanism.

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## 1. Introduction

Bone and joint degenerative inflammatory problems affect millions of people worldwide. These diseases often require surgery, including total joint replacement in case of deterioration of the natural joint. With the increasing life expectancy of the population there is a requirement for long-term performance of total hip replacement (THR) joints. Also, the number of young patients who present hip damage due to severe sport injuries or accidents is increasing. Since materials currently in use undergo degradation after 10–15 years of use, it is expected that these patients may have more than one THR during their lifetime. But revision surgeries involve complex reconstruction, take long time, are difficult for the patient and have a low success rate. Therefore, the development of a THR solution with a high long-term survival rate is mandatory. The goal is to minimize material loss, subsequent osteolysis and component loosening. This is a real challenge for materials that are to be used in the THR and nowadays ceramic materials are at the forefront to address this issue.

Ceramics generally have good biocompatibility and when compared to metals, they cause reduced osteolysis [1] and negligible ion content release [2]. Alumina ceramics, in particular the homologous tribopair Al<sub>2</sub>O<sub>3</sub>-on-Al<sub>2</sub>O<sub>3</sub>, are surgeons' first choice for patients younger than 50 years [3,4]. The low wear rate and reduced risk of osteolysis are the reasons for this preference; however, alumina is a

brittle material and has a small but persistent probability of fracture. A high rate of catastrophic alumina inlay failure has been reported [5]. To deal with this problem, zirconia-toughened alumina (ZTA) composites are the most recent solution available in the market (BioloX-Delta from Ceramtec), bearing with improved fracture toughness decreasing the ceramic fracture rate and wear [6].

Another well-known ceramic material for structural applications is silicon nitride (Si<sub>3</sub>N<sub>4</sub>), which presents a marked fracture toughness value of 10 MPa m<sup>1/2</sup> [7] higher than 6.5 MPa m<sup>1/2</sup> from ZTA [8]. Indeed, Si<sub>3</sub>N<sub>4</sub> ceramics were far-seeing proposed by Zhou et al. [9] as a structural material for articular implants, and in the last decade by others [7,10–17]. A finite element analysis (FEA) study revealed the adequacy of silicon nitride for hip resurfacing (HR) surgeries due to its mechanical reliability, specifically a lower long-term failure probability of silicon nitride compared to alumina is predicted [14]. As the probability of fracture is correlated with the mechanical strength, the in-vivo reliability is expected to improve by using silicon nitride-based ceramics for femoral heads and acetabular components. This property is as important as the wear resistance of both the femoral head and acetabular liner components for the success of the THR joints.

The wear resistance of Si<sub>3</sub>N<sub>4</sub> ceramics is highly improved with diamond coatings grown by a chemical vapor deposition (CVD) method [18–20]. The Si<sub>3</sub>N<sub>4</sub> ceramic ensures the best adhesion properties to CVD diamond coatings [21] due to the low thermal expansion coefficient mismatch and grain-to-grain epitaxial relationships between diamond crystals, assisting on the interface strength [22]. Nanocrystalline diamond (NCD), in particular, provides an ultra-hard surface with very low wear rates (10<sup>−9</sup>–10<sup>−8</sup> mm<sup>3</sup> N<sup>−1</sup> m<sup>−1</sup>)

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measured in ball-on-plate tests of NCD-on-NCD pairs in physiological simulated media [23]. Previous studies have also demonstrated the excellent biological behavior of NCD films with no cytotoxic effects [24,25]. For this set of reasons, in a recent review on alternative materials for articulation in total joint replacement by Sonntag et al. [26], NCD is considered a promising solution for improving wear resistance, among other coatings.

The present work describes a new proposal of a tribopair material for articular joint prosthesis, namely to be used in the total hip replacement (THR): all-nanocrystalline diamond coated silicon nitride ceramic hip joints, comprising of an NCD coated  $\text{Si}_3\text{N}_4$  ceramic femoral head working against an NCD/  $\text{Si}_3\text{N}_4$  acetabular liner. The hip simulator wear testing behavior corresponding to the initial one year routine activity of a patient (1 million cycles) is discussed.

## 2. Experimental

Acetabular liner and femoral head specimens of 28 mm of nominal diameter of silicon nitride ( $\text{Si}_3\text{N}_4$ ) bulk material were fabricated according to the powder technology process described elsewhere [17]. NCD coatings of about 10  $\mu\text{m}$  in thickness were grown onto the surface of the acetabular liner and head components by the hot filament CVD method, under deposition conditions already reported [17]. The coated specimens were then manually polished using soft cloths impregnated with a sequence of diamond abrasive suspensions and colloidal silica. The external surface of the acetabular liner samples was clamped by press fit into appropriately fabricated back metal shells, which were manually cemented with surgical cement (compound material based on polymethyl methacrylate) into suitable holders. The femoral heads were attached by press fit into their respective holders using a replica of the conical neck of a conveniently manufactured metal stem. Fig. 1 shows the appearance of the acetabular liner and the head specimens mounted in the respective holders. A total of three pairs of specimens were tested.

A 6-station hip joint simulator (H52-6-1000 AMTI) was employed for the wear test, all the tribopairs being mounted in the anatomical vertical position. The loading cycle, angular motions and lubrication were in accordance to the recommendations of the ISO 14242-1 standard [27]. A physiologic loading pattern with a maximum load of 3000 N and minimum load of 300 N was applied axially through the central axis of the acetabular liner-head. The three angular motions – internal/external rotation (IR/OR), flexion/extension (F/E) and abduction/adduction (AB/AD) – were synchronized at 1 Hz of oscillation frequency. The angular length values were 2° IR, 10° OR, 25° F, 18° E, 4° AB and 7° AD. Sterilized alpha-calf serum with 30 g/L of protein content (Vitrocell, batch number 007/12) was used as the lubricant. In order to minimize bacterial growth into the serum and precipitation of calcium phosphate on the bearing surfaces, 0.2 wt% of sodium azide and 20 mM of ethylene-diaminetetraacetic acid (EDTA) were added to the lubricant. All tribopairs were involved by a plastic bag (Fig. 2) that keeps the bearing surfaces lubricated, also having a system that allows closed loop fluid recirculation at 37 °C with minimization of evaporation. At 0.5 million cycle (1/2 Mc, first step) of test the acetabular liners and the heads were removed from the simulator and cleaned for the wear analyses, followed by restarting testing with fresh lubricant, taking care to keep the position of the tribopairs. The wear test lasted 1 million cycles (1 Mc) considering that a hip joint implant in a patient under normal routine activities experiences  $10^6$  cycles each year in average [28,29].

The wear was assessed by weighing the specimens after careful cleaning, using a high precision analytical balance (MSA225S-OCE-DI Sartorius, resolution of 0.01 mg). The volumetric wear in  $\text{mm}^3$  was calculated by dividing the weight loss by the NCD material theoretical density (3.515  $\text{g}/\text{cm}^3$ ). The linear wear (wear thickness in  $\mu\text{m}$ ) was estimated considering that the worn area is within a spherical cap that

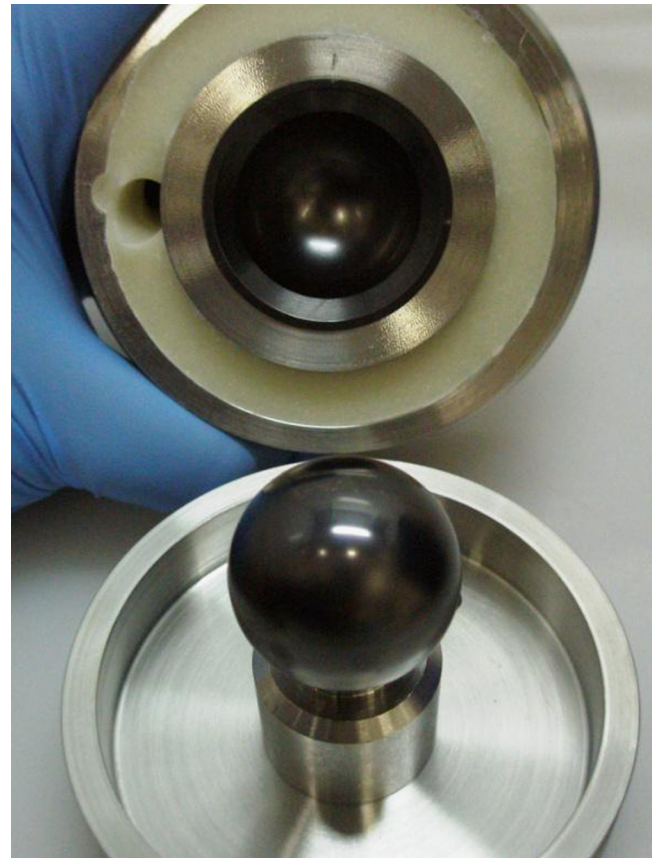


Fig. 1. General view of the femoral head and acetabular cup made of NCD coated silicon nitride.

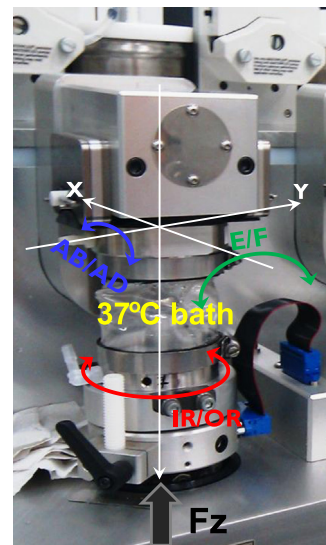


Fig. 2. Arrangement of the tested pair of materials (inside the plastic bag) in the hip wear simulator machine.

wears out homogeneously. The surface topography was assessed using a contacting profilometer (PGI830 Taylor Hobson) with a 2  $\mu\text{m}$  radius diamond tip stylus. The measurements were performed in the F/E and AB/AD directions passing through the pole of the samples, being 4 measurements of 10 mm length in the acetabular liners and 2 measurements of 15 mm length in the femoral heads. The  $R_a$  roughness parameter was calculated with a cut-off length of 0.08 mm. The microscopic features of the surfaces were assessed by high resolution scanning electron microscopy (HRSEM, Magellan 400 Fei

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