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The effect of lubrication on the friction and wear of Biolox®*delta*

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ABSTRACT

The performance of total hip-joint replacements depends strongly on the state of lubrication in vivo. In order to test candidate prosthetic materials, in vitro wear testing requires a lubricant that behaves in the same manner as synovial fluid. The current study investigated three lubricants and looked in detail at the lubrication conditions and the consequent effect on ball-on-flat reciprocating wear mechanisms of Biolox®*delta* against alumina. Biolox®*delta*, the latest commercial material for artificial hip-joint replacements, is an alumina-matrix composite with improved mechanical properties through the addition of zirconia and other mixed oxides. Three commonly used laboratory lubricants, ultra pure water, 25 vol.% new-born calf serum solution and 1 wt.% carboxymethyl cellulose sodium salt (CMC-Na) solution, were used for the investigation. The lubrication regimes were defined by constructing Stribeck curves. Full fluid-film lubrication was observed for the serum solution whereas full fluid-film and mixed lubrications were observed in both water and the CMC-Na solution. The wear rates in the CMC-Na and new-born calf serum were similar, but were an order of magnitude higher in water. The worn surfaces all exhibited pitting, which is consistent with the transition from mild wear to severe or "stripe" wear. The extent of pitting was greatest in the serum solution, but least in the water. On all worn surfaces, the zirconia appeared to have fully transformed from tetragonal to monoclinic symmetry. However, there was no evidence of microcracking associated with the transformed zirconia. Nevertheless, AFM indicated that zirconia was lost preferentially to the alumina grains during sliding. Thus, the current study has shown conclusively that the wear mechanisms for Biolox®*delta* clearly depend on the lubricant used, even where wear rates were similar.

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1. Introduction

Total hip-joint replacements (THRs) account for the largest proportion of orthopaedic surgery. Since their introduction, the lifetime of THRs has been greatly improved with time, and can now commonly last over 10–15 years [1,2]. However, the increasing number of younger and more active patients and the small but still significant occurrence of clinical failure (such as fracture and aseptic loosening) means that there is still a challenge to develop new biomaterials that are more damage-tolerant than the existing materials.

Modern commercial artificial hip-joint ball-in-socket combinations usually comprise metal (such as Co–Cr–Mo alloy), polymer (such as ultrahigh-molecular-weight polyethylene, UHMWPE) and ceramic (such as alumina) combinations. Since the first introduction of alumina-on-alumina THR was made in the 1970s, its high wear resistance and excellent biocompatibility make it an ideal material for THRs. Although some other ceramics (such as zirconia) were developed as alternative bearing materials, alumina THR has been the most widely used over the last 30 years. First introduced in the 1970s, more than 3.5 million alumina components have been implanted worldwide. However, poor performance of alumina-on-alumina THRs was observed early on, in particular a high fracture rate in vivo, which restricted its development worldwide. With the concerns related to fracture in ceramics, substantial improvements in microstructure have been made by controlling the processing to achieve medical-grade THR products according to ISO 6474. A major step forward was the use of hot isostatic pressing (HIP), which substantially increases the density of the component. In addition, small additions of MgO led to a smaller and more uniform grain size. This led to the so-called third generation of commercial alumina products, introduced in 1994, e.g. Biolox®*forte* produced by CeramTec AG, Germany. These have become increasingly popular THRs.

The lifespan of a ceramic-on-ceramic THR offers substantial improvements over other THRs, although concerns over component fracture led to a delay in the introduction of alumina-on-alumina THRs in the USA until recently. Retrieved implants from aseptic loosening reveal a distinctive localized region of high wear, known as "stripe wear" [3–7], associated with surface intergranular fracture. While the mechanisms leading to stripe wear are still a

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matter of debate, it has been shown that stripe wear occurs only where there is a micro-separation between femoral head and acetabulum during the walking cycle, leading to impact stresses [8]. The nature of stripe wear, and the concerns over component fracture, have driven the development of tougher ceramics. This has led to the development of the fourth-generation product, Biolox®*delta*, a zirconia-toughened alumina ceramic nanocomposite produced by CeramTec AG, Plochingen, Germany. This alumina-based ceramic composite has been successfully implanted and has offered outstanding performance in the last 8 years due to several reinforcing principles.

In vivo studies are the most convincing and authoritative method of obtaining data on long-term performance and the failure of hip prostheses. However, in vitro studies of artificial joints are carried out as the only method for systematically studying the performance of materials for THRs and avoiding uncontrollable variables such as patient size and their differing personal habits, etc. One of the problems in assessing a material's performance is accurately reproducing in vivo conditions during in vitro testing in order to fully understand the material response. Synovial fluid is a lubricant for natural cartilage bearings. Healthy synovial fluid exhibits typical non-Newtonian shear thinning characteristics, with medium to high viscosity [9], and promotes lubrication mechanisms spanning both full fluid-film and boundary lubrication [1]. Therefore, for in vitro tests it is necessary to control the lubrication conditions to be as close as possible to that in vivo. One difference is the inability to exactly replicate synovial fluid with an equivalent laboratory lubricant. New-born calf serum or bovine serum is the most widely used lubricant in joint simulation tests, giving a similar protein response to synovial fluid. New-born calf serum solution with 20 gl^{-1} protein is normally regarded as within the physiological range of the synovial fluid [10]. 25 vol.% new-born calf serum is the most widely used lubricant for artificial joint simulations [8,11]. Although new-born calf serum solution is commonly used, it cannot exactly replicate the friction conditions or the chemical interaction between the prosthetic material and the lubricant. 1 wt.% CMC-Na solution has also been used and compared with serum solution since it has similar rheological properties to synovial fluid [12,13]. In addition, according to ISO 6474, ultra pure water was also selected as a lubricant for a base line comparison. The ionic properties of water are known to affect wear behaviour and since ultra pure water is virtually free from impurities the electrical and chemomechanical effects [14] on the ceramic surface can be minimized.

On the basis of this, the current study focused directly on the reciprocating sliding wear behaviour of Biolox®*delta* in three commonly used lubricants, namely ultra pure water, 25 vol.% new-born calf serum solution and 1 wt.% CMC-Na solution. This project was initiated with the following principal objectives:

1. to understand the effects of lubricant type (e.g. new-born calf serum) on wear behaviour of bio-ceramics for joint prosthetic applications;
2. to understand the effect of adsorption of the lubricant into the surface, and how it affects near surface damage accumulation mechanisms;
3. to understand the role of the various microstructural features of Biolox®*delta* on wear, for example the zirconia particles.

2. Materials and methods

2.1. Materials used

The material used in the present study was Biolox®*delta*, which is the latest member in CeramTec's Biolox® family. It is an alumina-matrix composite with improved mechanical properties through the addition of zirconia and other mixed oxides, namely yttrium oxide, chromium oxide and strontium oxide. For the reciprocating sliding wear tests in the present work, the Biolox®*delta* specimens were manufactured into a disc-shape, 13 mm in diameter and 0.7 mm thick.

Surface roughness is clearly a key variable in lubrication studies. In order to ensure minimal contribution from roughness and to ensure consistency from test to test, the best possible surface finish was produced. This was achieved through grinding with silicon carbide papers and finishing with diamond suspensions and finally colloidal silica, Silco®(MetPrep Ltd, UK). By this approach, a roughness, R_a, of ~5 nm was measured by AFM contact mode scanning on an 80 μm × 80 μm area.

2.2. Lubricants

The ultra pure water used was the HPLC Gradient grade water (Fisher Scientific, UK), which has a viscosity of 0.001 Pas. 25 vol.% new-born calf serum solution was made by diluting sterile new-born calf serum supplied by First Link (UK) Ltd with phosphate buffered saline (PBS) 1× (0.01 M). PBS is a salty solution containing sodium chloride, sodium phosphate and potassium phosphate. It is commonly used in biochemistry because it is non-toxic and isotonic to cells. PBS is used as biomolecule dilutent since it can structure water around biomolecules immobilized to the solid surface and this thin water film can prevent denaturing of biomolecules or conformational changes. In the current study, the PBS was made by dissolving PBS tablets (Sigma–Aldrich, UK) with ultra pure water (Fisher Scientific, UK). To avoid bacterial growth and problems with protein degradation of the serum, 0.1 wt.% sodium azide (Fisher Scientific, UK) was added. This serum solution had a viscosity of 0.0012 Pas. 1 wt.% CMC-Na solution was made by dissolving purified CMC-Na cellulose powder (Fisher Scientific, UK) in ultra pure water (Fisher Scientific, UK). The solution was stirred by magnetic stirrer for 48 h. The viscosity of this solution was measured as 0.024 Pas at a shear rate of 3000 s^{-1} and 0.05 Pas at a shear rate of ~100 s^{-1}.

2.3. Wear testing

Reciprocating sliding wear tests under different lubricated conditions at room temperature were preformed on a reciprocating ball-on-flat UMT tribometer (Center for Tribology Inc., USA). A high purity alumina ball with 4 mm diameter was used as the counter body. These alumina balls with consistent roughness (~5–8 nm) were obtained commercially from Oakwade Ltd, UK.

Multiple combinations of normal load and sliding velocity were used to construct a Stribeck curve. The normal load in the wear test was 0.5, 1, 2, and 4 N. The maximum contact load corresponded to an initial Hertzian contact stress of ~3156 MPa, which fell rapidly during running-in. Such a value is inline with rim-contact stresses for ceramic-on-ceramic articulation [15]. The reciprocating motion was set at 500 rpm, 600 rpm and 700 rpm, which corresponded to frequencies of 8.333 Hz, 10 Hz and 11.667 Hz, respectively. An identical stroke length was preset as 10 mm for all the sliding wear tests. Specimens were ultrasonically cleaned with alcohol and then dried with compressed air. Before the test, the Biolox®*delta* specimen was mounted in the liquid chamber and submerged in the appropriate lubricant for at least 12 h. Fresh lubricant was transferred into the liquid chamber before each test. Sliding durations were set as 24 h, which has been shown to be well into the steady state wear regime. The lubricant in the chamber was changed every 12 h and the chamber temperature was well controlled to avoid protein degradation in serum solution-lubricated tests. The friction coefficient was measured using a high resolution force

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