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A wear simulation study of nanostructured CVD diamond-on-diamond articulation involving concave/convex mating surfaces

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Abstract

Using microwave-plasma Chemical Vapor Deposition (CVD), a 3-micron thick nanostructured-diamond (NSD) layer was deposited onto polished, convex and concave components that were machined from Ti-6Al-4V alloy. These components had the same radius of curvature, 25.4mm. Wear testing of the surfaces was performed by rotating articulation of the diamond-deposited surfaces (diamond-on-diamond) with a load of 225N for a total of 5 million cycles in bovine serum resulting in polishing of the diamond surface and formation of very shallow, linear wear grooves of less than 50nm depth. The two diamond surfaces remained adhered to the components and polished each other to an average surface roughness that was reduced by as much as a factor of 80 for the most polished region located at the center of the condyle. Imaging of the surfaces showed that the initial wearing-in phase of diamond was only beginning at the end of the 5 million cycles. Atomic force microscopy, scanning electron microscopy, Raman spectroscopy, and surface profilometry were used to characterize the surfaces and verify that the diamond remained intact and uniform over the surface, thereby protecting the underlying metal. These wear simulation results show that diamond deposition on Ti alloy has potential application for joint replacement devices with improved longevity over existing devices made of cobalt chrome and ultra-high molecular weight polyethylene (UHMWPE).

Introduction

The design of a replacement joint implant is critical to its long-term function, with wear of the joint articulating surfaces being of particular concern. The majority of artificial joint replacements employ a cobalt chrome (CoCr) alloy convex surface rotating inside a concave ultra-high molecular weight polyethylene (UHMWPE) insert. This has proven to be the most effective wear couple to date in terms of resistance to bulk fracture. However, clinical evidence shows that the surfaces of retrieved CoCr components exhibit scratches and roughening[1] that can significantly increase wear of polyethylene[2]. Polyethylene wear with associated osteolysis and implant loosening have been identified as the primary causes of long-term failure in joint replacements such as those for the hip and knee [3–4]. In order to reduce the number of revision surgeries, substantial improvements in the longevity of prosthetic joints require a system-wide approach including the development of advanced

bearings. Monolithic ceramic implants (e.g. zirconia) are limited due to the concerns of brittle fracture resulting from age-induced phase transformations [5]. Clearly, the combination of high hardness, high toughness, and low friction is important in implant design, as is the mechanical integrity of the surface over many years of *in vivo* service.

The wear of implants has been extensively studied, *in vivo* and *in vitro* [6–8]. Most wear studies can be classified into three categories: metal-on-UHMWPE, metal-on-metal, and ceramic-on-ceramic. Cobalt chrome alloys are often used for the metal in these devices. Typically, wear of hard articulating surfaces reveals an initial wearing-in period of high surface material loss before establishing a significantly reduced linear wear rate[9]. This is due to the grinding away of surface asperities until the surfaces are mated such that the load is being applied uniformly.

The aim of the study was to create surfaces similar in size and function as those of the natural temporomandibular joint (TMJ) and simulate the articulation of the joint in a simple fashion to demonstrate their suitability in an implanted device. The challenge was to produce clinically-relevant conditions in terms of implant geometry and load/displacement for the articulating surfaces.

Prior work using the NSD surface applied to titanium alloy substrates showed that when used in a pin-on-disk type configuration (under dry wear conditions) substantial wear and delamination of the diamond coating occurred [10]. However, this configuration placed the contact area under a large contact stress, approximately 2.8 GPa, leading to a deformation of the base metal and delamination of the film on the curved surface. The yield strength of the underlying metal is only 830 MPa which is not enough to support the large contact stress without deforming. Thus the hard, brittle film could not be expected to survive this scenario in the long term. Another study using a condyle (convex) and fossa (concave) arrangement, where the surfaces were curved but still not matching in curvature, showed much improved performance of the diamond surfaces [11]. However, this setup was also conducted under dry wear conditions, and relatively short (2.25×10^5 cycles) simulations were performed. Thus the next level of testing was to lubricate the surfaces with simulated body fluid and extend the simulation length, while making the surfaces more closely conforming to distribute the load.

The configuration used in the current study is an attempt to remedy this situation by matching the curvature of both surfaces so that the applied load is distributed evenly. In a perfect contact situation for the current configuration, the stress would only be 3.46 MPa, well below the yield strength of the metal. In order to determine an appropriate load for the wear test, ASTM standard F732-00 (2011) was chosen, even though this standard is for wear testing polymeric materials used in total joint prosthesis. It was chosen due to the lack of existing wear simulation testing standards for ceramic—on—ceramic devices. In this standard, the average contact stress should be 3.54 MPa; this value is specified for a sliding wear test using polymer on metal. Another specification in the standard is for a “hip-type” wear test, with a designated contact stress of 2–10 MPa. The configuration chosen for the current test, with a calculated contact area of approximately 65 mm² and a load of 225N provides a contact stress of 3.46 MPa. Therefore, the wear test performed in this study falls

very close to the specification for linear wear and is well within the range for the “hip-type” test. At least for small mouth opening movements, the conventional “ball in cup” geometry of the hip joint is similar to that of the TMJ.

Another rationale for choosing the 225N load for the wear simulator test follows from the work of Marks *et al* [12] where they measured the strain on the condylar bones of miniature pigs and then used the surface area of the condyles to calculate the load on the condyle surface. They estimated the load at 135N per condyle for mastication and 227N for stimulation (single muscle contraction). Alternatively, a method of approximation is to use the value for the bite force reported by Hylander [13] which is 196 N. Multiplying this value by 80% (the condylar reaction force is approximately 75–80% of the bite force [14]) gives a value of 157 N for the load on the condyle during biting. The configuration used in the current study creates a simplified approximation to the type of articulation seen in the TMJ for small mouth opening movements. Thus the selection of 225N load for the wear simulator test matches well or exceeds specified and published wear loads for similar tests and applications.

Materials and Methods

Wear simulation was performed with an AMTI (Boston, MA) Force-5 multi-axis testing machine. This machine is capable of producing movements through 5 degrees of freedom, measuring forces in three dimensions, and has a chamber for maintaining the test specimens in fluid at 37° C. Figure 1 illustrates the setup as modified for our testing. The standard load cell (for knee simulation tests) was replaced by the manufacturer with a lower range unit with a maximum force of 1000N to more effectively accommodate the loading range expected for TMJ implant testing. The wear couple can be enclosed within a chamber filled with simulated bodily fluid and held at body temperature to simulate an *in vitro* environment. Special fixtures were designed to hold the much smaller shapes used to simulate portions of the TMJ. The desired wear simulation parameters can be programmed into the machine by creating a load-motion waveform profile. The machine uses the feedback from the load cell to modify the motion either by trying to maintain a constant force or a constant motion. For the current study the desired motion was setup so that a constant force was applied to the wear couple along the vertical axis, and the upper component was rotated about an axis $\pm 15^\circ$ at a rate of 2 Hz. This produced a sliding distance per cycle of 53.2mm, which is close to the standard of 50mm (section A1.3.2.11, ASTM F732). This motion was meant to simulate the chewing action of the TMJ in a simple fashion, even though it neglects the sliding component of the natural TMJ motion.

The wear couples were milled from rod stock of wrought Ti-6Al-4V Grade 23 biomedical grade alloy. The parts consist of a ball and cup type configuration with the fossa being the cup component and the condyle as the ball component, as shown in Figure 2. The radius of curvature was 25.4mm on both parts. The articulating surfaces were machined to a tolerance of less than ± 0.00127 mm deviation from the radius of curvature and the surface roughness (R_a) was measured with a diamond stylus profilometer (ATS-032 at ATS Laboratories, Atlanta, GA) to be 180 nm.

These surfaces were then processed using a microwave plasma Chemical Vapor Deposition (CVD) system to deposit a 3–5 μm layer of NSD[15]. The NSD coatings are not phase-pure diamond, but consist of nanocrystalline diamond grains (10–100 nm) along with amorphous carbon to yield a measured hardness of 60–70 GPa [16]. The NSD can withstand substantial strain without hard elastic-to-brittle fracture occurring and has been shown to remain well adhered to titanium alloys under large coating/substrate interfacial strains[17–19]. The excellent adhesion of the diamond to the titanium alloy has been attributed to the formation of a thin layer of titanium carbide before the formation of the diamond layer[21].

Figure 3 shows a high resolution SEM image of a typical NSD coating. For comparison, Figure 4 shows AFM for microcrystalline diamond.

The AMTI Force 5 wear simulator was setup such that the wear couple was held in place by a rotating arm that holds the condyle. The lower, fossa component was fixed onto a stage that applies the vertical load from below via a hydraulic piston. The load cell is located inside the stage and measures the forces applied in three dimensions. The rotating arm and sample were aligned so that the surface of the condyle extends 25.4mm from the rotating axis of the arm. In this way, the surface of the condyle smoothly articulated against the fossa during the test.

An alignment protocol was developed for the initial setup of each mated wear couple. It was quickly found to be a critical part of the procedure, as a small misalignment of the wear couple will create uneven loading and extremely high stress concentrations. The samples were inserted into the fixtures of the simulator and visually aligned. A very small vertical load (approximately 5N) was applied to bring the surfaces in contact. A careful sequence of small horizontal shifts in all four directions (front/back, left/right) was used while observing a graph of the vertical load. The location whereby the load was minimized (corresponding to an optimum surface contact) was determined to be the correct center axis for the wear couple.

After preparing the wear samples for testing, the sample enclosure (the saline bag as seen in Figure 1) was filled with a simulated body fluid comprised of bovine serum diluted to a protein concentration of 23g/L [21], with an addition of 7.45g/L Ethylene-Diamine Tetraacetic Acid (EDTA), and 0.2 wt% sodium azide (per ASTM F732, section 5.2.5). This fluid was exchanged for a new solution every week, approximately every 0.5 million cycles.

A 1.3 million cycle wear test was conducted with a set of NSD coated condyle and fossa as an initial test of the machine and samples. The samples were checked each day and the load was increased throughout the test starting at 30N and finishing with 400N. Once it had been shown that the test samples could survive this high loading condition, a full wear test was conducted. A 5-million cycle test on a new NSD-coated sample set was carried out with a constant load of 225N. The wear couples were cleaned after removal from the system using a sequence of ultrasonic baths, first in water and enzymatic detergent (Biz[®]), then in acetone, followed by methanol. An inhomogeneous film, as shown in Fig. 5, formed during the wear simulation process on the diamond surfaces near the contact area. This type of film has been reported in other wear simulation studies [9] with the layer most likely consisting

of protein deposits. It was found that this film did not dissolve even when immersed in an enzymatic cleaner. The film was effectively removed after gentle polishing of the surface using a cotton ball moistened with colloidal silica. Optical microscopy, Scanning Electron Microscopy (SEM), 3D optical imaging using white light interferometry, as well as AFM was performed to image the NSD coating (after cleaning) and to gauge the extent of surface wear.

Results

The current study demonstrates a critical step toward the successful development of a new TMJ replacement device. The curved articulating surfaces showed minimal polishing of the NSD coating after 5 million cycles under a constant force of 225N; this represents a load higher than that predicted in normal loading conditions on the natural condyle. Also of note is that under a very high loading condition of 400N for 172,800 cycles, the surfaces did not show any damage or delamination (Figure 6). The wear simulation demonstrated that the coatings are well adhered and exhibit no signs of cracking or flaking. None of the samples delaminated or showed signs of wearing through the thickness of the coating. In fact, the longest wear simulation experiment did not create enough wear to bring the surfaces into full conformal sliding contact as shown in Figure 5.

In a typical wear study the samples are measured at specified intervals for weight loss, thereby providing the wear volume as a function of the number of cycles. In these results the volume of wear produced over the 5 million cycles was so small that it was not possible to measure the wear as a weight loss. As a result a number of imaging techniques were used to evaluate the surface after the wear simulation.

Optical imaging of the condyle contact area before, during, and after 5 million cycles is shown in Figure 7. Even after 5 million cycles only light polishing of the diamond surface occurred. It was expected that the full surface of the condyle component would slide across the surface of the fossa, but it can be seen that there was some mismatch in the radius of curvature of the two components, with the condyle having a slightly smaller radius. This change in shape is thought to be a result of the CVD deposition process, where the Ti alloy is expanding after heating and cooling by a small amount during the diamond growth process. The alloy is absorbing hydrogen and carbon as a result of the CVD process. Currently experiments are being conducted to account for the mismatch by accurately measuring the amount of expansion of the material and then altering the initial curvature to account for the dimensional change.

SEM images of select areas of the diamond surface on the fossa piece after the 5-million cycle wear simulation are shown in Figure 8. Figure 8(a) shows an area outside the contact zone, this was chosen to show the texture of the surface as deposited, i.e. an area with no wear. Figure 8(b) is in a region where there was partial contact. It can be seen that the asperities in this region are partially worn down and smooth areas can be seen in between rougher patches that have not been abraded. Figure 8(c) shows the area from the center of the fossa where the surfaces were in full contact and most of the asperities were worn down, but not completely smooth as is the condyle surface in the center.

AFM was performed on various areas of the diamond wear surface after 5 million cycles to determine the extent of material loss during wear. Figure 9 shows AFM of (a) an uncoated Ti-6Al-4V condyle along with several images (b)–(e) of the NSD-coated condyle at various positions near the apex (after software leveling to account for substrate convexity). Line profiles are also provided to indicate the height variation from NSD surface asperities. The transition in morphology from (b) to (e) of Fig. 9 demonstrates progressively worn regions where (b) represents a region of no wear (i.e. as-grown NSD) since it was not in direct contact with the fossa. Region (e) represents the maximum wear at the central apex as it articulated continuously with the fossa. Due to the curvature of the condyle and nature of motion in the wear simulator, regions (c)–(d) experienced less contact. Asperities on the NSD film are observed to have gradually worn down revealing a smoother surface closer to the central apex. As expected, the progressively worn areas result in decreasing peak-to-valley surface roughness values, starting with the unworn NSD coating (b) of 160 nm. Closer to the apex, the corresponding average peak-to-valley values are (c) 80 nm, (d) 40 nm, and (e) 2 nm. Since there are no remaining surface asperities at the central apex region (d), we can estimate a lower limit of wear depth for the condyle to be approximately 160 nm. Once the wear process reaches the level of (e), it is expected that the wear rate would slow considerably because of increased surface contact area and correspondingly reduced contact pressure. Nevertheless, continued wear would occur. Figure 10 shows an AFM image of shallow ($S_a=25\text{nm}$) wear grooves near the center of the condyle. These are thought to be a result of the fossa still having asperities that are being ground down and are creating grooves in the condyle.

Another useful roughness parameter is the surface developed interfacial area ratio (Sdr) [see ISO 25178, section 4.3.2 for a mathematical definition] which expresses the increment of the interfacial surface area relative to the area of the projected (flat) x,y plane. A completely flat surface would return $Sdr = 0\%$. The Sdr values for regions (b), (c), (d), and (e) are 5.0%, 4.1%, 0.94%, and 0.00011%, respectively. These values also indicate the progressive removal of surface asperities from off-center apex regions to the central apex region, resulting in a nearly perfect flat, polished surface at the central apex.

Quantitative 3D analysis of the surface was performed with a TLA-Tencor MicroXAM-800 optical profilometer with a 10X objective. The profilometer was operated in phase shift interferometry mode to obtain the smoothest surface measurements. The main features observed in the center where the contact between the two articulating surfaces was the greatest were linear wear tracks approximately 25nm deep and circular grooves which are machining marks from the metal fabrication process. Figure 11 shows the center area of the condyle after the 5-million cycle wear test. The wear grooves are the same as those imaged by AFM and both imaging methods show the same average depth of grooving.

Another confirmation of the existence of the diamond surface after wear testing was to use Raman spectroscopy. A presence of the 1332 cm^{-1} peak in a Raman scan would show that diamond still exists on the surface. Using an excitation wavelength of 532nm, scans were taken of the central wear region and areas of the surface that did not experience wear (see Figure 12). This revealed that for even the most worn region (Fig. 9(e)), the NSD coating was intact and unmodified from its original (unworn) structure.

Conclusion

A wear simulation using an articulating motion meant to mimic the movement of the human TMJ was conducted with NSD coated titanium pieces. The wear couple consisted of two titanium alloy pieces with curved conforming, polished surfaces onto which a nanostructured diamond surface had been applied. This wear couple was tested for 5-million cycles at 2Hz cycling frequency and 225N vertical load and revealed very little wear over the duration of the test. From AFM data on the condyle it is estimated that a minimum of 160nm of wear was produced during the “wear-in” phase in the central region of greatest contact. Shallow linear wear grooves were imaged in the surface of the condyle using 3D profilometry and AFM. It was observed that the fossa component was not completely worn smooth and this explains why the condyle has the wear grooves approximately 25nm in depth. Raman spectroscopy confirmed that the NSD coating remained intact and was unmodified in structure from its as-grown state. The surfaces did not have a uniform distribution of wear as evidenced by the difference in surface roughness after testing, going from 80nm roughness (σ_{p-v}) in the edge region to 40nm mid-radius to 2nm in the center (see Table 1). This indicates that the total area of actual sliding contact was less than expected based on estimates of conformal matching. As a result, the region of contact did not appear to have transitioned fully from the wear-in stage—where the highest parts of the two bearing surfaces grind away at each other—into a full-contact, steady state wear mode. Due to the fact that the wear rate during the “wear-in” phase is always found to be higher than the steady state wear rate, it is expected that the diamond wear rate will decrease as more of the surface is polished down.

The survival of the NSD surface with no delamination, cracking, or exposure of the underlying metal surface indicates that the diamond surface is a good candidate for protecting the surface of implanted replacement joint devices under high wear conditions, such as the TMJ. By testing the curved surfaces under loading conditions similar to that of the natural TMJ, we have shown that the diamond surface is ready to move to the next stage of testing, using a more complex rotating and translating profile movement in the simulator, once the surfaces can be made to match more closely in shape. This type of movement will more closely reflect the anatomical TMJ motion.

Future work will include improving the starting surface roughness which ideally would translate into less abrasion and wear, thus extending the life of the implant even further. Other motions to be tested include high load/short translation cycling, which would replicate the type of loading that occurs during grinding of teeth. This type of loading is thought to contribute to the deterioration of the natural TMJ and thus is a situation for which the implant must be tested.

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Figure 1.
Wear simulation setup showing fluid housing and wear specimens.

Figure 2.
NSD-coated wear couple specimens; fossa on left and condyle on right.

Figure 3.
Hi-res SEM surface morphology of our NSD coating showing granular structure.

Figure 4. AFM image of (a) typical microcrystalline diamond film and (b) typical nanostructured diamond film

Figure 5.
The fossa as removed from wear simulator, with material built-up on the surface. The scale bar is 2mm.

Figure 6. Center area of sample tested to 400N during wear testing. Dark areas are polished diamond which is highly reflective.

Figure 7.
Surface of condyle after 5 million cycles of wear. The dark areas are polished and highly reflective.

Figure 8.

Selected areas of the fossa surface after wear testing. (a) is near the edge where no wear occurred, (b) is in the area where partial contact was made between the surfaces, (c) is where the most wear occurred, in the center.

Figure 9.
AFM data from condyle surface after wear test.

Figure 10.
AFM image of area with shallow linear wear grooves. Surface roughness $S_a=25\text{nm}$.

Figure 11.
3D profilometer image of center of condyle after wear simulation. The shallow wear tracks can be seen going from left to right.

Figure 12.

Raman spectral intensity from diamond surface on condyle after wear test at: (a) center wear area, (b) transition zone off center, (c) edge (unworn area).

Table 1

Roughness values for condyle subjected to 5 million cycle wear test

	Uncoated condyle (center)	NSD coated condyle (center)	Far edge wear area	Mid-radius wear area	Center of wear area (apex of condyle)
Roughness (σ_{p-v}) (nm)	49	160	80	40	2