

A Wear Evaluation of Ultra High Molecular Weight Polyethylene (UHMWPE) against Nanostructured Diamond-Coated Ti-6Al-4V Alloy



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Reducing the wear of joint replacements would increase the lifespans of both the replacement and the patient. In this study, the wear of ultra-high molecular weight polyethylene (UHMWPE) against nanostructured diamond (NSD)-coated titanium alloy (Ti-6Al-4V) and uncoated cobalt-chromium alloy (CoCr) hemi-cylinders was compared to determine if the NSD coating results in less volumetric wear of polyethylene (PE). A wear simulator was used with a gait cycle to mimic the knee joint, but with an axial force ranging from 30N to 700N during the cycle. Both tests ran for 1.5 million cycles while immersed in bovine serum. The roughness and volumetric wear of the NSD-coated alloy were greater than the non-coated control sample. No significant differences in the PE surface structure hardness were observed for either wear couple, as measured using Raman spectroscopy, X-ray diffraction, and nanoindentation. Although the roughness of the PE (worn by NSD-coated alloy) decreased faster than that of the control PE surface (worn by CoCr), the as-deposited surface roughness of the NSD coating was about three to four times higher than that of the starting CoCr surface. These results suggest that a much higher abrasive wear occurred for the NSD-PE couple due to the high NSD surface roughness, which also accounted for its inferior wear performance. Due to the higher initial surface roughness of the NSD-coated hemi-cylinder, the wear of the NSD-PE couple is greater than the non-coated couple, indicating that this combination would not increase the lifespan of a replacement joint.

INTRODUCTION

The number of total knee replacements in the US has doubled in the last ten years (Salomon et al., 2010; Weinstein et al., 2013). In addition, the average age of patients receiving a total knee replacement (TKR) is decreasing (Weinstein et al., 2013). It is becoming increasingly important to determine how to reduce wear and degradation of implanted joints in order to improve their long-term performance (Smith, Dieppe, Porter, & Blom, 2012). Most TKRs contain an ultra-high molecular weight polyethylene (UHMWPE) tibial component that articulates against a metallic alloy femoral component (Ritter, 2009). These pieces are often attached to the

bone with bone cement but this method of fixation has the potential to create inflammatory debris and wear-induced periprosthetic osteolysis (Ritter, 2009).

It is essential that TKRs be made to last longer than they do now, especially given the fact that the average human lifespan is projected to increase significantly over the next 100 years (Weinstein et al., 2013). In a 15-year survivorship study (Ranawat et al., 1993), only 70.6% of patients who weighed more than 80kg survived. If implant wear can be decreased, the life expectancy of people weighing over 80kg could potentially increase. Assuming humans continue getting TKRs around the age of 65 (Danilidis & Tibesku, 2012), the knee replacement must last at least 20 years, ideally 30. To enhance the lifespan of the knee replacement, the current focus has been on improving the UHMWPE spacer, which simulates the articular cartilage that allows for smooth movement of the femur and the tibia. It has been shown that polyethylene (PE) wear particles (Amstutz, Campbell, Kossovsky, & Clarke, 1992; Ritter, 2009; Teeter, Parikh, Taylor, Sprague, & Naudie, 2007) generated from the PE spacer causes osteolysis and possible loosening of the tibial plate (Willert, Bertram, & Buchhorn, 1990). The purpose of this study is to reduce this wear.

Third-body wear (the introduction of hard particles in the space between two articulating members), has been shown to increase the roughness of the cobalt-chromium (CoCr) femoral component and PE wear (Davidson, 1993; Lawson, Catledge, & Vohra, 2005; Pierannunzii, Fischer, & D'Imporzano, 2008; Wang & Essner, 2001). A study done by Wang and Essner (2001) showed that loose poly-methyl-methacrylate (PMMA) bone cement particles in

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the lubricant, in excess of 5g/L, adhere to CoCr femoral heads and lead to accelerated wear of the acetabular cups. In contrast, the attachment of PMMA particles to ceramic heads was much reduced, resulting in an UHMWPE wear rate that was independent of the concentration of the PMMA particles.

To minimize third-body and other mechanisms of wear from shortening the lifespan of a knee replacement, a nanostructured diamond (NSD) or amorphous carbon coating on the femoral component can be used (Amaral et al., 2007). The tribological benefits may be attributed to: (1) superior lubricating properties (more wettable, hence better able to maintain lubricant on the surface), (2) high hardness and (3) relative inertness of the material. These characteristics can provide a decrease in the coefficient of friction at the bearing surface, with less susceptibility to third-body wear and scratching, as well as less biological response to any debris generated by ceramic wear particles (Lawson et al., 2005; Zietz, Bergschmidt, Lange, Mittelmeier, & Bader, 2013). Recently, it has been shown that the lubricity of amorphous carbon arises from shear induced strain localization, which dominates the shearing process (Pierannunzii et al., 2008). This lubricity is characterized by covalent bond reorientation, phase transformation and structural ordering in a localized tribolayer region. A transition in movement from stick-slip friction to continuous slipping, with ultra-low friction, is observed due to gradual clustering and layering of graphitic sheets in the tribolayer. This enhanced lubricity potentially offered by a variety of carbon-containing coatings may reduce wear in total joint prostheses.

In a previous pin-on-disk study (Hill et al., 2008), we showed that a NSD-coated Ti-6Al-4V disk surface resulted in a factor of two less wear of the PE pin when compared to a non-coated CoCr disk. However, this study was not representative of the gait cycle experienced by the knee joint. In the present study, we compare the effects of wear of an NSD-coated titanium alloy hemi-cylinder to a non-coated CoCr control using a multi-axis wear simulator to more closely mimic the knee joint. We hypothesize that the NSD-coated hemi-cylinder will cause less wear compared with the non-coated hemi-cylinder due to more favorable lubricity and reduced friction conditions.

METHODS

For wear-testing, an AMTI Force 5 machine (Advanced Mechanical Technology, Inc. Watertown, MA) was used by applying a cyclic vertical load ranging from 30N to 700N on the flat PE samples shown in Figure 1. The tests followed ISO (International Organization for Standardization) standard 14243-3 (ISO, 2014). However, the axial force was scaled down during the gait cycle from a maximum of 2600N (as stated in the standard) to 700N, due to limitations of our load cell capacity. Flexion movement (rotation of flexion arm around a horizontal axis), anterior and posterior movement, and rotation of the stage were incorporated into the cyclic waveform representative of the knee gait cycle, according to the ISO standard. Approximately 1.5 million cycles at 1Hz were performed on each PE sample using the uncoated CoCr and

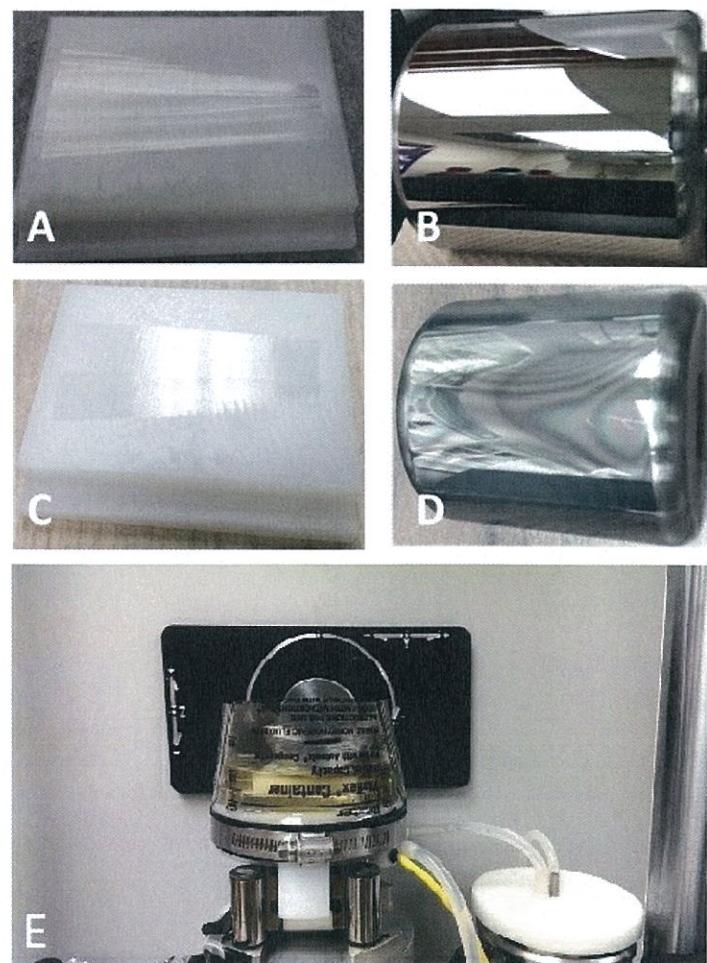


Figure 1. Experimental setup. A) UHMWPE sample with wear from CoCr counterface, B) CoCr counterface, C) UHMWPE sample with wear from diamond coated counterface, D) diamond coated counterface, E) in situ experiment in bovine serum.

the NSD-coated hemi-cylinder. For the uncoated upper piece, the CoCr hemi-cylinder was attached to a rotating flexion arm using a thin layer of bone cement. For the NSD-coated piece, a titanium hemi-cylinder of the same dimensions was used as the substrate for coating. This hemi-cylinder was attached to the flexion arm using two stainless steel bolts.

To determine the effect of the wear testing, four PE samples were measured: two controls (soaked for an equivalent time in bovine serum but not wear-tested) and two wear-test samples. The wear-test samples were imaged using atomic force microscopy (AFM) and weighed every 250 thousand cycles. All samples were kept completely submerged in a bovine serum mixture at 37°C for the duration of the 1.5 million cycles. The serum mixture (consisting of 0.2% w/v sodium azide [0.8g], 20mM EDTA [2.98mL], 100mL bovine serum, and 300mL deionized water) was replaced every 750 thousand cycles. To counteract evaporation, 100mL of deionized water was added every day to the machine reservoir. The

control samples were kept in a sealed container, without adjusting volume.

Weighing Samples

To obtain the weight of the wear-test samples, a cleaning procedure was followed based on ASTM (American Society for Testing and Materials) F732 "Standard Test Method for Wear Testing of Polymeric Materials Used in Total Joint Prostheses" (ASTM 2011). The samples were retrieved from the machine and rinsed with water. Extraneous particles were removed with lens paper. Next, samples were sonicated for 15min in 100mL of tap water and 1mL of liquid cleaner. After rinsing in deionized water, sonication procedure was repeated for an additional five minutes in 100mL of deionized water. Following sonication in deionized water, the samples were soaked in methanol bath for three minutes. Next, the samples were placed in a desiccator for 30min. Finally, samples were weighed and imaged to determine changes in mass and surface roughness.

Atomic Force Microscopy (AFM)

AFM imaging was done to determine how the surface roughness of the PE samples was changing over the 1.5 million cycles. Imaging was performed using close-contact mode at a scan rate of 0.45Hz with 256 points per line. Four AFM images were taken in various regions of the PE sample with scan areas of 10 μm^2 and 30 μm^2 . Surface roughness values were obtained using Scanning Probe Image Processor (SPIP) 5.1.1 (Image Metrology A/S, Hørsholm, Denmark) and MS Excel. Optical microscope images of the wear samples were also taken at the start and end of the 1.5 million cycles. The wear-test samples and control samples were weighed at the same intervals to account for mass gain from serum absorption. The overall change in roughness of the PE samples was recorded. AFM was also done on the surface of the hemi-cylinders before and after wear.

Volumetric Wear

In order to calculate volumetric wear, the mass of the wear-test sample was corrected by subtracting the mass absorption of the control sample from that of the wear-test sample measured at the same interval. The initial mass measurement (M_0) was subtracted from each consequent measurement (M_m) to acquire the mass difference (M_d). The change observed, true mass (M_t), of the control sample was then subtracted from the change in the mass of wear-test sample. The wear volume (mm^3) was calculated from M_t using the density of UHMWPE (0.9363kg/cm³).

Nanoindentation

Nanoindentation was performed on the PE samples using a Berkovich diamond tip (nominal radius 50nm) to a depth of one μm . Indentation was done before and after wear testing to detect any changes in surface hardness or elastic modulus, potentially as a result of structural transformations.

X-ray Diffraction

X-ray diffraction ($\lambda = 1.54154\text{\AA}$) was used on the PE sample surfaces to further examine potential effects of wear, such as phase

transformations or texturing, from articulation against the NSD-coated and non-coated alloy.

Raman Spectroscopy

Laser Raman spectroscopy ($\lambda = 514.5\text{nm}$) was performed on the control and wear-test samples to evaluate possible structural transformation (such as disordering of carbon bonds), as measured in the wavenumber range from 1000 to 1800cm⁻¹.

RESULTS

In this study, we evaluated two samples: a control UHMWPE sample worn against CoCr (Sample 1) and a test UHMWPE sample worn against NSD-coated Ti-6Al-4V alloy (Sample 2). Nanoindentation performed on both samples before and after wear did not show a significant change in either hardness or Young's modulus. Compared to the UHMWPE/CoCr couple, the NSD/UHMWPE couple produced higher volumetric wear. The volumetric wear of Sample 1 was 1.20mm³ after 250k cycles and reached a value of 3.72mm³ at the end of the 1.5 million cycles (Fig. 2). By comparison, the wear of Sample 2 after 250k cycles was 6.29mm³, which was already almost twice as much as the maximum wear volume of Sample 1. Note that the volumetric wear trends of these two samples are different. Sample 1's volumes tapered off in the final measurement intervals whereas Sample 2 showed a consistent amount of wear occurring at each interval. Using a parabolic curve fit, the best fit gives an R^2 value of .8476 for Sample 1. The linear fit of Sample 2 has an R^2 value of .9945. Overall, the volumetric wear of Sample 2 is higher and more consistent than the wear of Sample 1.

The initial average roughness value of Sample 1 (PE worn by CoCr) was lower than the initial average roughness of Sample 2 (PE worn by the NSD-coated alloy) (Fig. 3). Sample 1's initial roughness was $177.21 \pm 42.23\text{nm}$ while Sample 2 had the initial roughness of $260.64 \pm 69.74\text{nm}$ for an AFM scan area of 30 μm^2 (Fig. 3). The final roughness of Samples 1 and 2 were $27.32 \pm 5.67\text{nm}$ and $11.13 \pm 3.42\text{nm}$, respectively. The overall roughness of Sample 1 decreased by 85% while the overall roughness of Sample 2 decreased by 96%. For an AFM scan area of 10 μm^2 , Sample 1 had a decrease of 80% in overall roughness, which is 8% lower than that of Sample 2. The roughness of both PE samples dropped by more than 90% within the first 500 thousand cycles (as measured from an AFM scan area of 30 μm^2). The outlier at approximately 1 million cycles for the roughness of Sample 1 had an average value of 128.16nm with a relatively large standard deviation obtained from four separate measurements, the least of which was 51nm.

No measurably significant change in surface roughness was detected from either the uncoated CoCr or NSD-coated hemi-cylinders before and after wear (Fig. 4). However, it should be noted that all roughness values for the CoCr hemi-cylinder are below 8nm, while those of the NSD-coated alloy are about a factor of three larger. Finally, Raman spectroscopy revealed that no significant change in PE carbon bonding occurred after wear (Fig. 5).

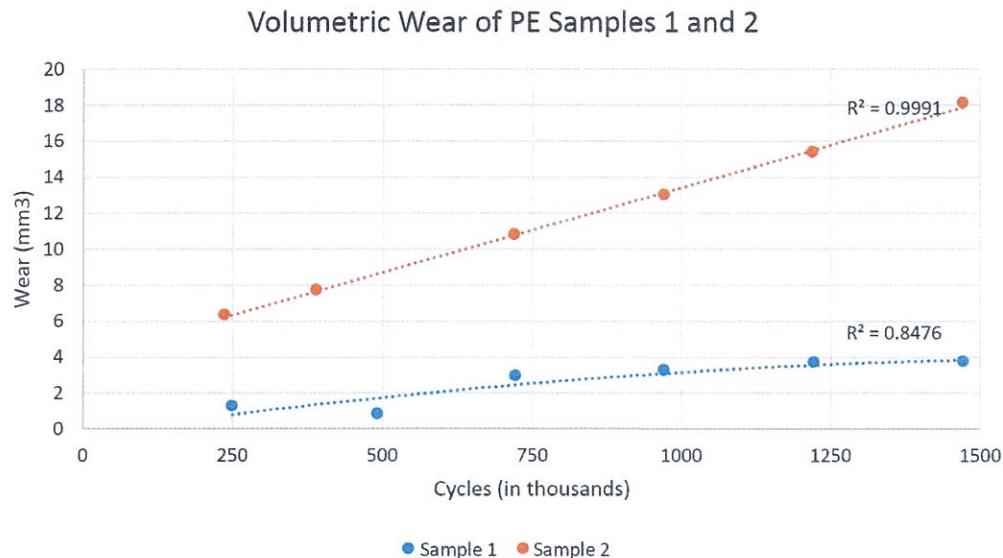


Figure 2. Volumetric wear of Samples 1 and 2. Sample 1 (worn by CoCr): The starting wear is around 1.2mm^3 while the ending wear is 3.7mm^3 . The R^2 value of a parabolic line of fit is .8476. The rate of wear decreases as the number of cycles increases. Sample 2 (worn by Ti alloy): The starting wear is around 6.3mm^3 while the ending wear is 18mm^3 . The R^2 value of a linear line of fit is .9991. The rate of wear seems to stay consistent as the number of cycles increases.

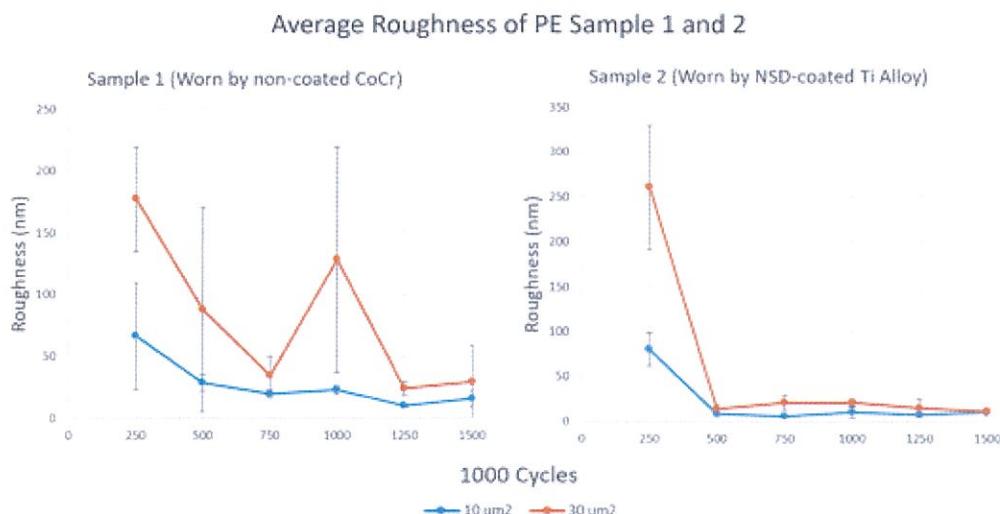


Figure 3. Average roughness of PE Samples 1 and 2. The starting roughness for both samples at areas of $10\mu\text{m}^2$ and $30\mu\text{m}^2$ is higher than the final roughness. The Sample 1 roughness value on approximately 750 thousand cycles is larger than expected. However, the error is very large as well. Four samples per data point yields errors bars that are less than 1 standard deviation (SD). Sample 1: Data from 250k ($10\mu\text{m}^2$), 500k ($30\mu\text{m}^2$), and 750k ($30\mu\text{m}^2$) cycles had a roughness range greater than 1 SD but less than 2 SD 's. Sample 2: Data from 250k ($30\mu\text{m}^2$) cycles had a roughness range greater than 1 SD .

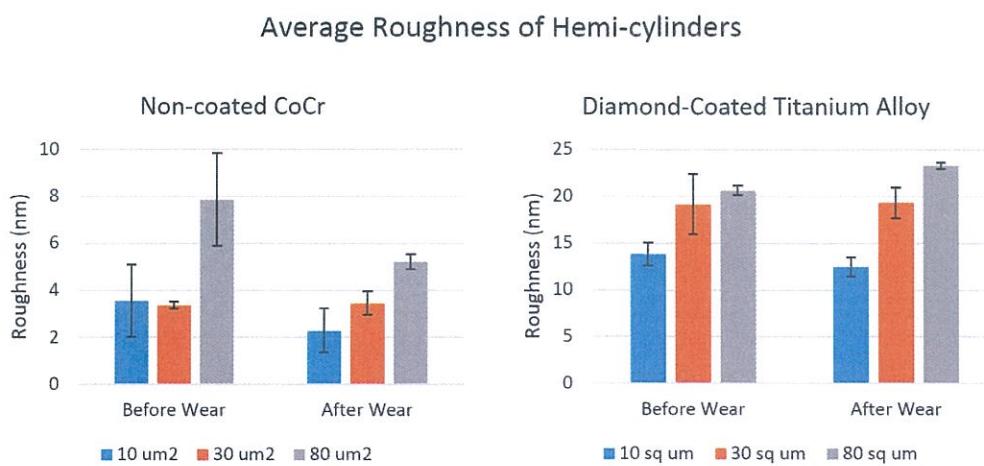


Figure 4. Average roughness of CoCr and titanium alloy hemi-cylinders. Both the non-coated CoCr and diamond-coated Titanium alloy generally show that, as the area being imaged increases, so does the average roughness. Both “before wear” and “after wear” show this same trend. For the non-coated CoCr hemi-cylinder, the “before wear” error bars ($10\mu\text{m}^2$ and $80\mu\text{m}^2$) ranged greater than 1 SD .

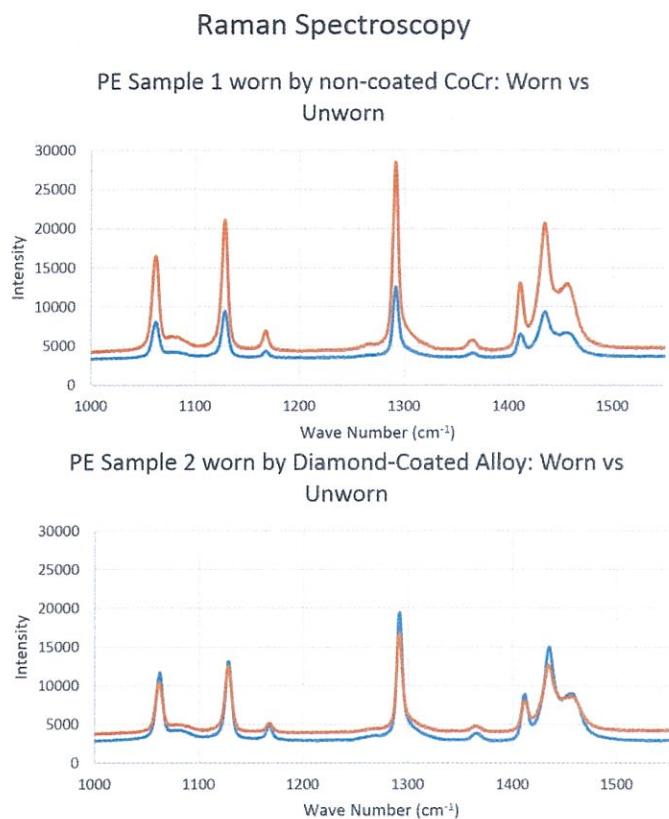


Figure 5. Raman spectroscopy of PE Samples 1 and 2. Raman Spectroscopy peaks remained unchanged for both PE samples.

DISCUSSION

With the number of people receiving total knee replacements growing, as well as an overall increase in lifespan, the need for a longer-lasting total knee replacement is becoming more urgent. Our results indicated that the wear effect of an NSD-coated titanium hemi-cylinder on a PE sample was greater than the wear from a non-coated CoCr on a PE sample. The UHMWPE/NSD resulted in a smoother surface on the PE sample.

The volumetric wear and AFM roughness data both showed that more wear occurred for the UHMWPE/NSD couple compared to the control UHMWPE/CoCr couple. Given that the starting surface roughness of the NSD coating was three to four times higher than that of the CoCr surface, it suggested that excessive abrasive wear occurred for the UHMWPE/NSD wear couple. Our result showed the opposite result from a previous pin-on-disk study (Hill et al., 2008), which found that less wear occurred for the UHMWPE/NSD couple compared to an UHMWPE/CoCr couple. In that study, the NSD coating was smoother by a factor of five and the loads/displacements were much less clinically relevant. In order to test the potential benefits of an NSD-coated counterface against UHMWPE for a total joint replacement, future efforts will need to be directed toward decreasing the NSD surface roughness to a value at least as small as the starting bare alloy. Otherwise,

abrasive wear may dominate the wear mechanisms.

Figure 2 shows that, if extrapolated, the curved trend seen in the volumetric wear of Sample 1 can be expected to continue increasing at a slower rate. However, when observing the volumetric wear of Sample 2, no such trend is seen. Instead, a nearly linear trend is observed along with a high R^2 value of .9991. If this trend were continued, it would most likely increase linearly, as it did for the past 1.5 million cycles.

Based on the results of this study, the NSD-coated alloy, with its relatively high surface roughness compared to the CoCr alloy, would cause unacceptable wear of the PE inserted in an artificial joint, leading to a high probability of early implant failure. As shown in Figure 2, the volumetric wear of the control PE (Sample 1) appears to drop off near the end of the 1.5 million cycles. This trend is not seen with the test PE (Sample 2). Instead, the wear rate is consistent for the entire 1.5 million cycles. The average roughness for PE Sample 2 (Fig. 3) shows that the roughness rapidly decreases within the first 500 thousand cycles and remains nearly unchanged after that. Therefore, we cannot assume that the mass loss is proportional to a decrease in surface roughness. Instead, it is more likely that abrasive wear caused by the NSD surface initially removed asperities from the UHMWPE (whose initial roughness was several hundred nanometers) and then continued to wear this surface at a constant rate. Since the NSD surface is a factor of three to four rougher than the CoCr, one would expect abrasive wear to be higher for this wear couple. For the UHMWPE/CoCr couple, the apparent plateau in wear may be explained by improved lubricity at the metal/polymer interface as the polymer surface roughness drops. Though the roughness changed greatly throughout the wear testing, both Raman spectroscopy and x-ray diffraction (XRD) showed no significant changes between the control and test PE samples.

An outlier in measured surface roughness from Sample 1 was observed at the 4th measurement interval (near 1 million cycles). At this point in the wear-test, the roughness is not uniform throughout the contact surface areas. One of these less-smooth areas could have been chosen for collecting these data rather than the typical smoother locations used in the other experiments. The four average roughness values obtained for this measurement had a very large range. It is not clear why these measurements resulted in such large spread. Since the surfaces of the alloys were not of the same roughness at the beginning, this could have also affected how much polyethylene was worn during the 1.5 million cycles. To maintain consistency, both alloy surfaces should be either polished or chosen to have the same roughness. Given the limitations of this study, the overall wear of polyethylene from the non-coated alloy was nearly a factor of five less than that of the NSD-coated alloy.

The NSD-coated hemi-cylinder resulted in nearly five times more PE wear than that from the non-coated CoCr hemi-cylinder while the average PE roughness decreased much faster for Sample 2 (involving the NSD counterface) than for Sample 1 (involving the non-coated CoCr counterface). This suggests a more aggres-

sive abrasive wear-in period in the beginning due to the higher surface roughness of NSD. To determine how significant the wear rate is, more PE samples should be tested. This will help ensure reproducibility as well as reduce random errors.

The surface of the NSD-coated hemi-cylinder itself showed no appreciable changes in average roughness before and after wear. For both control and test PE samples, no change in mechanical properties (hardness or elastic modulus as measured by nanoindentation) or in carbon bond structure (as measured by Raman spectroscopy) were detected. Both XRD and nanoindentation should be done on the sample worn by the NSD-coated hemi-cylinder in the future. These techniques could help detect changes in the PE crystallinity as well as possible alignment of the PE fibers. Although this data suggests that an UHMWPE/NSD couple is inferior, the influence of starting surface roughness must be considered as a limiting factor. All counterface surfaces should have similar and low initial roughness values to ensure reproducibility. This could be achieved through polishing. We hope to achieve a similar surface roughness for both counterfaces by improving the NSD coating process. The NSD-coating needs to be have reduced roughness and improved consistency over relatively large surface areas. In the future, reduction of NSD coating surface roughness should be a primary goal for a better comparison to the control wear couple.

Although this study evaluated an UHMWPE/NSD couple, an alternative couple that could lead to more promising results would involve NSD/NSD. In this way, the NSD-coating may be expected to minimize abrasion while acting as a barrier to prevent leaching of potentially toxic heavy metal ions from the substrate. The applications for this coating are more suitable for artificial hip joints (which also employ hard-on-hard bearings). Perhaps, for a ceramic-on-ceramic hip replacement, an NSD coating could even further increase the longevity of implants (Bhatt & Goswami, 2008; Hill et al., 2008; Ranawat et al., 1993).

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