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# Evaluation of wear suppression for phospholipid polymer-grafted ultra-high molecular weight polyethylene at concentrated contact



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#### ABSTRACT

The wear resistance of cross-linked polyethylene grafted with poly(2-methacryloyloxyethyl phosphorylcholine) (PMPC-grafted CLPE) and unmodified CLPE loaded by an alumina roller were evaluated in a simulated synovial fluid (0.5 wt% hyaluronate solution containing 30 vol% fetal bovine serum). To evaluate the wear depth distribution within the wear scar, we compared the projected area of wear and non-wear for indents on polyethylene surfaces produced by a triangular pyramidal indenter and estimated the local wear depths on the basis of the changes in the projected area. The worn depth of PMPC-grafted CLPE was almost a third lower than that on CLPE.

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#### 1. Introduction

Deterioration of the function of synovial joints, in diseases such as osteoarthritis and rheumatoid arthritis, decreases the capacity for daily motion and causes severe pain, but it is possible to recover physical function and relieve pain by replacing the joint with an artificial joint composed of biocompatible artificial materials. Most commonly, artificial joints are composed of ultra-high molecular weight polyethylene (UHMWPE) combined with ceramics (alumina or zirconia-toughened alumina) or metals (cobalt-chromium or titanium alloys). However, there is a serious problem: long-term use of an artificial joint induces osteolysis, which is attributed to a xenobiotic response of macrophages to micro-wear particles of UHMWPE [1] and often causes joint loosening, requiring revision of the artificial joint. To overcome this problem, several treatments, such as gamma-ray radiation cross-linking [2–4], changing the combination of materials used for the sliding surface [5], and addition of vitamin E [6,7], have been used, resulting in some cases in significantly improved wear resistance and attenuation of osteolysis. However, these measures do not address the root of the problem.

Therefore, we considered that it is necessary to understand the superior lubrication mechanism of the natural joint and duplicate its functioning as far as possible in an artificial joint to enhance lubrication and thereby improve the wear resistance of the artificial joint.

It is known that there is a nanometer-scale protective film layer containing phospholipid at the surface of the articular cartilage in synovial joints and that the layer protects the articular surface and improves the lubrication mechanism [8]. In recent years, Moro et al. demonstrated that cross-linked UHMWPE (CLPE) grafted with nanometer-scale phospholipid polymer (poly[2-methacryloyloxyethyl phosphorylcholine]; PMPC) (PMPC-grafted CLPE) functioned like the natural protective film covering articular cartilage and provided excellent performance in artificial hip joints, showing low friction and minimal wear after 3–70 million cycles in hip simulator tests [9,10]. Additionally, PMPC-grafted CLPE had such excellent biocompatibility that its micro-wear debris did not cause bone-resorptive responses [9]. Thus, PMPC-grafted CLPE has been successfully used in clinical applications in artificial hip joints with good geometrical conformity [11,12]. Further, as the next stage of development, a knee simulator study of a PMPC-grafted CLPE tibial insert placed against a cobaltchromium alloy femoral component showed that PMPC grafting dramatically decreased the generation of CLPE wear particles [13]. However, the dependence of the volume of generated wear particles

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http://bio.mech.kyushu-u.ac.jp/SPR/contact.html.

on the contact pressure applied in these simulator tests have yet to be clarified

In our previous study [14], we performed a reciprocating friction test for a PMPC-grafted CLPE pin placed against a sapphire plate at 2.5 MPa using various lubricants, with in situ visualization of the fluorescently stained PMPC layer. We reported that damage to the PMPC layer was accelerated and the friction coefficient was increased in a fetal bovine serum (FBS) solution, but deterioration was mitigated by adding hyaluronic acid to the FBS solution. The mixtures of hyaluronic acid and FBS solution closely duplicate human synovial fluid. In tests using the simulated synovial fluid, the PMPC layer exhibited superior hydration lubrication ability like that of articular cartilage in synovial fluid.

In this study, the wear resistance of the PMPC layer was examined to evaluate the effectiveness of the application of PMPC grafting to artificial joints with concentrated contact load such as total knee prosthesis. To examine wear behavior at higher contact pressure than that used in the previous test (2.5 MPa), we selected 20 N loading with a maximum Hertzian pressure of 19.5 MPa and mean pressure of 13.0 MPa on the surface of elastic contact. However, the actual maximum contact pressure decreased to 7–8 MPa as creep deformation progressed and the wear area of the CLPE increased. Therefore, we evaluated the changes in rubbing surfaces at 3.0–7.0 MPa for 20 N loading. A sliding velocity of 94.2 mm/s was selected, which corresponds to the sliding speed during the stance phase of a knee joint replacement.

In this study, we adopted the indentation techniques [15] to evaluate the degree of wear by comparing the projected areas of indents in wear and non-wear zones. It was hard to adopt the most popular methods, such as the profilometry of entire wear scar or the gravimetric analysis of specimen, in this case, because of the presence of hard pick feed direction on the specimen surface and a small amount of wear in each wear test. To permit the simplification of measurement, furthermore, we tried to estimate the wear depth from difference in the projected area of indent in the optical microscope image.

#### 2. Materials and methods

#### 2.1. Roller-on-flat tester

Fig. 1 shows the roller-on-flat tester for the sliding test. The upper roller specimen was motor driven, and the sliding velocity was controlled by driver unit adjustment. The roller specimen shaft was supported by bilaterally located rolling bearings in the wear test. The lower flat specimen was fixed in a lubricant bath. The vertical load was applied by hanging the required weight on the other side of a lever.

### 2.2. Upper specimen

The upper specimen was a polycrystalline alumina ceramic roller with a spherical surface of 30 mm in radius and 10 mm in width. The arithmetic average roughness (Ra) of the upper specimen in the sliding direction was about 0.01  $\mu$ m. Before the wear tests, the specimens were subjected sequentially to ultrasonic cleaning with solutions of 1 vol% Triton X-100, pure water, and 99.5% ethanol for 30, 30, and 15 min, respectively, and then dried in a vacuum at 60 °C for 2 h.

#### 2.3. Lower specimens

#### 2.3.1. CLPE specimen

Compression-molded bar stocks of UHMWPE (GUR1020 resin; Quadrant PHS Deutschland GmbH, Vreden, Germany) were irradiated with a 50 kGy dose of gamma rays in a  $N_2$  gas atmosphere and

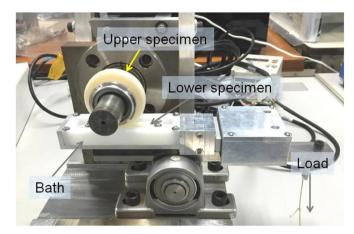


Fig. 1. Photograph of a roller-on-flat tester. The bearing box on the near side of the shaft is removed.

annealed at 120 °C for 7.5 h in  $N_2$  gas to facilitate cross-linking. Hereafter, these materials will be referred to as CLPE. The CLPE samples were then machined from bar stock after cooling. The specimen geometry was rectangular (60 mm  $\times$  20 mm) with a thickness of 4 mm. The rubbing surfaces of the CLPE specimens were polished, resulting in the Ra in the sliding direction of about 0.2  $\mu$ m.

#### 2.3.2. PMPC-grafted CLPE specimen

The CLPE specimens were immersed in acetone (Wako Pure Chemical Industries, Ltd., Osaka, Japan) containing 10 mg/mL benzophenone (Wako Pure Chemical Industries, Ltd.) for 30 s and then dried in the dark at room temperature to remove the acetone. The MPC was industrially synthesized using the method reported by Ishihara et al. [16] and purchased from NOF Corp. (Tokyo, Japan). The MPC was dissolved in degassed pure water to a concentration of 0.5 mol/L. Subsequently, the benzophenone-coated CLPE specimens were immersed in the MPC aqueous solution. Photo-induced graft polymerization was conducted on the CLPE surface using UV irradiation (UVL-400HA ultrahigh-pressure mercury lamp; Riko-Kagaku Sangyo Co., Ltd., Funabashi, Japan) with an intensity of 5.0 mW/cm<sup>2</sup> at 60 °C for 90 min; a filter (model D-35; Toshiba Corp., Tokyo, Japan) was used to restrict the passage of UV light to wavelengths of  $350 \pm 50$  nm [17]. After polymerization, the PMPC-grafted CLPE specimens were removed, washed with pure water and ethanol, and dried at room temperature. These specimens were then sterilized by a 25 kGy gamma-ray source in a N2 gas atmosphere. Finally, Ra in the sliding direction of the PMPC-grafted CLPE specimen was about 0.2 µm. Fig. 2 shows schematic diagrams and a Transmission Electron Microscope (TEM) image of obtained PMPC-grafted CLPE sample.

To quantify the local depth of the wear scar generated by the wear test, indents were prepared on the rubbing surface of the lower specimen at intervals of 300 µm (as appropriate interval to suppress the harmful influence of concave dent) using a dynamic ultramicro hardness tester (DUH-211; Shimadzu Corp., Kyoto, Japan) equipped with a 115° triangular pyramid indenter controlled so that the maximum applied force was 250 mN, the minimum force was 1.96 mN, the loading rate was 70.07 mN/s, the load holding time was 5 s, and the unload holding time was 5 s. Thereafter, the depth of indent recovered as viscoelastic polymer. To appropriately evaluate wear for the same initial indent depth in wear test, the polyethylene specimens with indents were used after sufficient viscoelastic recovery in deformation had been attained for 3 days.

Before the wear tests, the lower specimens were subjected to ultrasonic cleaning in 1 vol% Triton X-100 and pure water for 5 min each and subsequently immersed in 99.5% ethanol for 15 min. The specimens were then dried in vacuum at 60  $^{\circ}$ C for 2 h.

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