



Amine formation on the surface of porous calcium-phosphate artificial bone by low-pressure pulsed plasma polymer deposition

^{1,2}Anjar Anggraini Harumningtyas, ¹Tomoko Ito, ³Miroslav Michlichek, ¹Satoshi Sugimoto, ³Lenka Zajickova, ⁴Joe Kodama, ⁴Takashi Kaito, and ¹Satoshi Hamaguchi

¹ Center for Atomic and Molecular Technologies, Graduate School of Engineering, Osaka University, 2-1 Yamadaoka, Suita, Osaka 565-0871 Japan

²Center for Accelerator Science and Technology, National Nuclear Energy Agency of Indonesia (BATAN), Jalan Babarsari Kotak Pos 6101 ykbb Yogyakarta 55281, Indonesia

³Central European Institute of Technology, Brno, Czech Republic

⁴Department of Orthopaedic Surgery, Osaka University Graduate School of Medicine, 2-2 Yamadaoka, Suita, Osaka, 565-0871, Japan

Amine-rich plasma polymerization was performed on the surface of porous calcium-phosphate (hydroxyapatite [HA] or beta tricalcium phosphate [β -TCP]) artificial bone with a low-pressure plasma system. The aim of this study is to form amine groups on the surface of HA or β -TCP based porous artificial bone to improve its biocompatibility and osteogenic abilities. Experiments were performed with a bipolar pulsed plasma system [1-3] with a CH₄/N₂/He mixed gas at a pressure of 70 Pa [4]. The applied bipolar voltage was about ± 1.2 kV and the pulse duration was about 1 μ s for each positive or negative pulse. The pulse frequency was 5 kHz, so that the time elapses between a positive pulse and the subsequent negative pulse was 100 μ s. Calcium-phosphate artificial bone samples were placed on the grounded metal electrode and exposed to the plasma for 30 min. The porous artificial bone samples used in this study had interconnected pores with a porosity of 72%-78%. The average pore diameter was 150 μ m and the average diameter of a circular passage connecting two adjacent pores was 40 μ m. X-ray Photoelectron Spectroscopy (XPS), Fourier-transform infrared (FTIR), and Ellipsometry measurements showed that polymer deposition successfully took place on the artificial bone surface and the thickness of the film deposited on a Si substrate under the same conditions was about 40 nm. It was found that polymer deposition took place even on the inner surfaces of pores at least down to the depth of 2 mm from the sample surface. The elemental composition of N atoms of the deposited film was 10 ~ 15 % and found to be stable at least over a couple of months when the plasma treated artificial bone was kept in a desiccator. The derivatization using 4(trifluoromethyl)benzaldehyde (TFBA) showed that the surface concentration of primary amine groups over nitrogen atoms was 20 %. Effects of sterilization on plasma treated artificial bone by autoclave, ethylene oxide gas (EOG), and gamma ray radiation were examined. Surface characterization of deposited polymers and their biological effects will be also presented.

References

- [1] S. Sugimoto, M. Kiuchi, S. Takechi, K. Tanaka, S. Goto, *Surface and Coatings Technology* **136**, 65 (2001).
- [2] S. Takechi, S. Sugimoto, M. Kiuchi, K. Tanaka, and S. Goto, *Surface and Coatings Technology* **136**, 69 (2001).
- [3] Z. Donko, *et al.*, "Modeling characterization of a bipolar pulsed discharge" submitted (2020).
- [4] M. Michlichek, *et al.*, "Molecular dynamics simulation of amine groups formation on polystyrene surfaces," submitted (2020).