Low-temperature atomic layer deposition (ALD) of strontium oxide on poly(ether-ether-ketone) (PEEK) for lumbar cage implants

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Abstract: The deposition of strontium oxide (SrO_x) was studied with combination of thermal atomic layer deposition (ALD) and plasma enhanced ALD (PE-ALD) on poly(ether-ether-ketone) (PEEK) as surface modification of lumbar cage implants. Process temperature must be kept relatively low not to deform PEEK during the process. Direct deposition of SrO_x on PEEK was compared with that on titanium (Ti) or TiO_x covered PEEK. Deposited films were analyzed with ellipsometry or reflectometry, X-ray photoelectron spectroscopy (XPS), atomic force microscopy (AFM), and scanning electron microscopy (SEM). The release of Sr from the deposited film immersed in phosphate-buffered saline (PBS) to the solution was evaluated by inductively coupled plasma-atomic emission spectroscopy (ICP-AES).

Keywords: SrO_x, thermal ALD, PEALD, PEEK

1. Introduction

Poly(ether-ether-ketone) (PEEK) has been used as an artificial spinal cage material since 1998 because its elastic modulus is sufficiently close to that of human cortical bone [1][2]. However, its biocompatibility needs to be further improved. Recently, atomic layer deposition (ALD) and plasma-enhanced ALD (PE-ALD) have been used to modify surface properties of artificial bone implants made of PEEK. It has been reported that thermal ALD-based Al_2O_3/TiO_2 thin films exhibit good adhesion [3][4] and reduce moisture permeability of PEEK [5]. Thermal ALD also has been used to deposit TiO_2 on PEEK with various (e.g., smooth, rough, and porous) topographies. For example, porous PEEK covered with ALD-based TiO_2 showed strong osseointegration [6]. PE-ALD was also used to deposit TiO_2 thin films on PEEK implants and

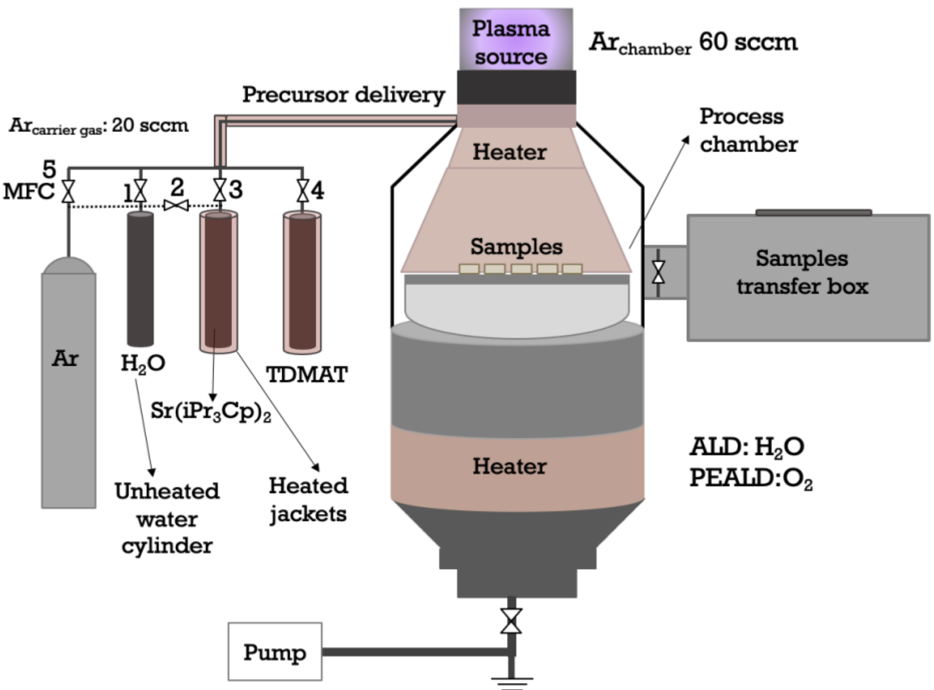

Fig 1. Schematic of thermal ALD of SrO_x process cycle

found to improve their osteogenic and bioactive properties [7].

Strontium (Sr) is known as the promising substances that can enhance the biocompatibility of artificial bone materials [8]. Sr has a dual effect of stimulating the osteoblast function and inhibiting the osteoclast function and can be used for osteoporosis treatment [9,10]. There has been a limited number of studies related to strontium deposition by ALD for bone implant applications. Thus, in this study, we attempt to establish ALD techniques of strontium oxide (SrO_x) on PEEK as a potential method to coat PEEK spinal cages with SrO_x. The advantage of ALD is its atomic-level controllability of the film growth that enables conformal coating. However, the deposition temperature must be limited to a maximum of 250°C because PEEK can significantly deform at temperature higher than 250°C.

2. Deposition films of SrO_x and SrO_x/Ti

In this study, thin cylindrical PEEK samples with rough surfaces were used as substrates. Each sample has a diameter and a depth of 12mm×2 mm. Si (100) wafers were used as control substrates to optimize thermal ALD/PE-ALD process parameters. The deposition experiments were carried out with Cambridge Nano Tech Fiji F200, the schematic diagram of which is shown in Fig.1. The PEALD is generated by inductively coupled plasma (ICP) 13.56 MHz. Bis(tri-isopropyl-cyclopentadienyl) strontium 98% ($Sr(iPr_3Cp)_2$) (Strem, Massachusetts, USA) was heated at 150°C. The Ar booster was necessary to assist the strontium precursors to go into the chamber. The process cycles of ALD and PE-ALD used in this study are summarized in Figs. 2 and 3.

First, the conditions for SrO_x ALD were optimized on the silicon substrate. Second, direct deposition of SrO_x on PEEK was attempted. However, it was found that SrO_x hardly deposited on a PEEK surface directly. Therefore, we coated PEEK with ALD-based titanium oxide (TiO_x) or magnetron-sputtered metallic Ti films prior to SrO_x ALD on the PEEK sample. The deposited films were analyzed with X-ray photoelectron spectroscopy (XPS), atomic force microscopy (AFM), and scanning electron microscopy (SEM). The thicknesses of deposited SrO_x films were determined by ellipsometry or reflectometry. The amount of Sr that can dissolve from deposited SrO_x thin film immersed in phosphate-buffered saline (PBS) to the solution was evaluated with inductively coupled plasma-atomic emission spectroscopy (ICP-AES). The optimal ALD conditions for SrO_x deposition at low surface temperatures (< 250°C) were obtained in this study.

3. Acknowledgments

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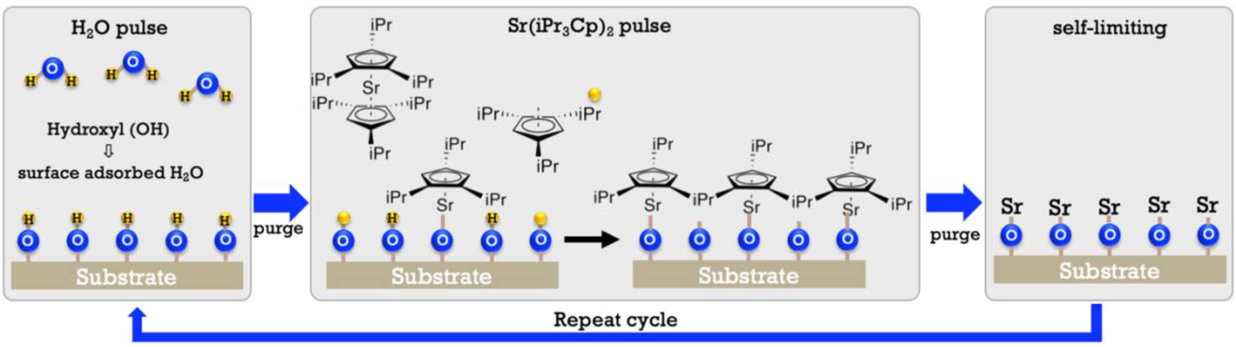

Fig 2. Schematic of thermal ALD of $SrOx$ process cycle

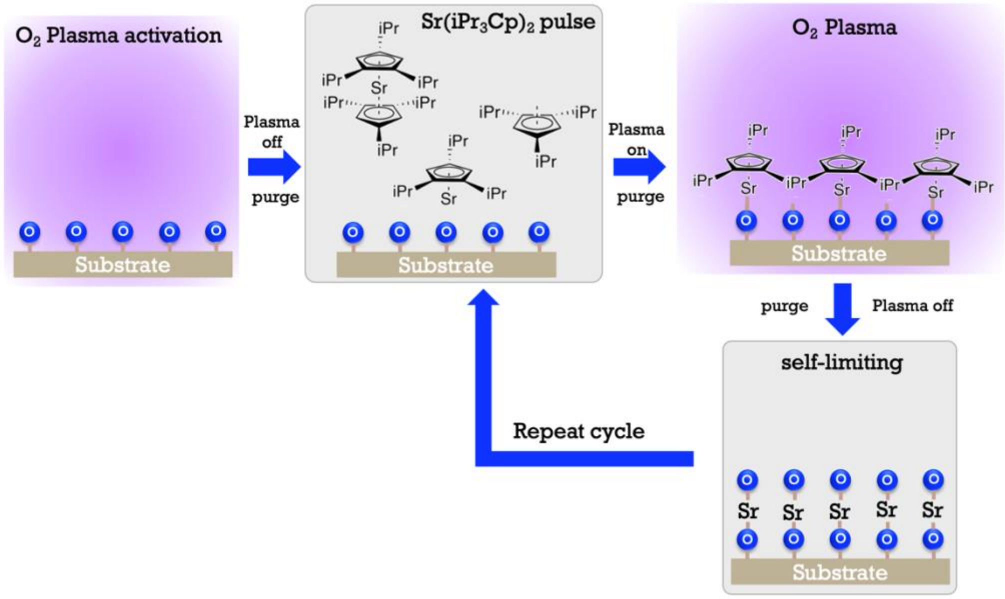

Fig 3. Schematic of PEALD of SrO_x process cycle