RESEARCH PAPER

Histomorphometric Evaluation of Porous Structures Polyether Ether Ketone (PEEK) Implant Coated with Nano Zirconium and Titanium Oxide Nanoparticles

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ABSTRACT

PEEK, a thermoplastic polymer with exceptional performance, is Polyether Ether Ketone. Moreover, PEEK surfaces are coated with titanium and its derivatives, TiO2 and ZrO, to improve their biocompatibility and bioactivity. Based on calcium phosphate, Human osseous material is most synthetically comparable to Hydroxy Apetite, a bioceramic that is commonly employed. sixty-four screws with machined surfaces were implanted in 32 adult male New Zealand white rabbits measuring 1.5–2 kg. Both the left and right tibia of each rabbit had an implant placed in it; one was used for testing and the other as a control. The animals were carified two and six weeks after implantation. The implants were split into two groups: experimental (32 titanium and zirconium oxide-coated peek implants) and control (32 peek uncoated implants). Based on the mean results, at the same time in week 6, the titanium oxide and zirconium oxide nanoparticles treated group had fewer osteoclasts than the control group (improve osteointegration) There was a statistically significant difference between the control and experimental groups in every healing phase, according to an analysis of all histomorphometric parameters. In this work, magnetron sputtering was used to thinly coat polyether ketone-based implants with titanium and zirconium to improve surface characteristics and promote osseointegration with implants.

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INTRODUCTION

Polyether Ether Ketone (PEEK), a high-performance thermoplastic polymer, is being used more and more in orthopedics [1] PEEK has a relatively low modulus of elasticity (3–5 GPa) in comparison to titanium (Ti; 102–110 GPa), more akin to the diaphysis of a bone [2–3]. Areas of high strain or stress at the contact between the implant and bone, which might affect bone repair, should not develop due to such a low modulus value. Furthermore, PEEK does not result in artifacts during magnetic resonance imaging (MRI) or

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computed tomography (CT) examinations. Implant surface characteristics have a significant impact on tissue response. In order to improve integration with bone tissue, PEEK surface modification may make it more conducive to osteoblast growth [4].

HA is a commonly used bioceramic based on calcium phosphate and is the most synthetically comparable to human bone mineral [5-30].

Furthermore, coatings made of titanium and its derivatives TiO_2 [26] and TiN are applied to PEEK surfaces to enhance their biocompatibility and bioactivity. In comparison to uncoated PEEK, Ti-coating significantly improved osteoblast adhesion and increased their proliferation, as Chang Yao et al. [6] showed. This work investigated the viability of coating polished screw-like PEEK substrates with thin layers (up to 100 nm) of Zr and Ti using direct current (DC) magnetron sputtering in an argon atmosphere.

Among the benefits of thin coatings is their ability to preserve the topography of uneven surfaces on sandblasted or acid-etched implants. Therefore, thin coatings may be employed to maintain the original morphology and structure even in porous materials that have been 3D printed. [7,8,27]

MATERIALS AND METHODS

Sixty four screw with machined surfaces were implanted in 32 adult male New Zealand white rabbits measuring 1.5–2 kg. Both the left and right tibia of each rabbit had an implant placed in it; one was used for testing and the other as a control. The animals were carified two and six weeks after implantation.

Using a magnetron sputtering machine in direct current (DC) mode and targets composed of titanium and zirconium with a working area of 190 cm^2, coatings were created for the surface modification of PEEK implants. It was determined that the power density was ((current_voltage)/ working area). The vacuum chamber was first drained to a 7×1-3 Pa pressure. Then, argon (Ar,99.9%) was poured into the chamber until the pressure within stayed between 0.7 and 0.9 Pa. Both targets were cleaned for ten minutes using a current of 2.0 A while a protective screen was placed over them. After that, a holder holding samples was used in lieu of the screen, and it was rotated by a motor

For both the Ti and Zr targets, the magnetron sputtering procedure was conducted at a current value of 0.2 A in order to prevent overheating and consequent damage to the PEEK samples. For Ti and Zr nanoparticles, the power density was 126.0 mW/cm^2 and 84.0 mW/cm^2, respectively.

The implants were split into two groups: experimental (32 titanium and zirconium oxide-coated(nano) peek implants) and control (32 peek uncoated implants), The sterilizing implants were inserted into 3 mm-diameter holes drilled in the rabbits' tibias. Coated implants were used on the

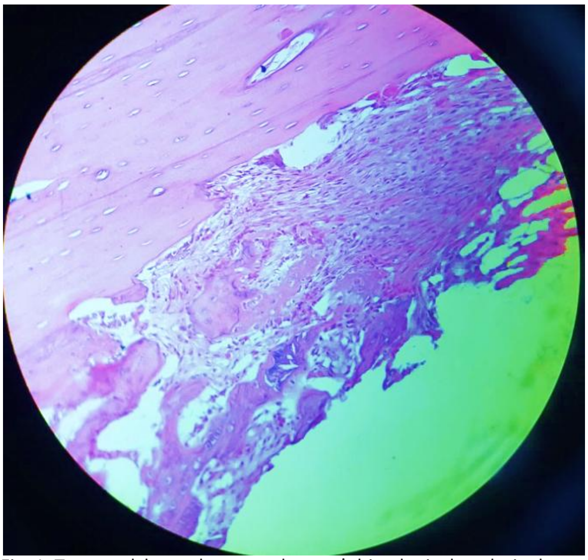


Fig. 1. Two week later, the control group's histological analysis shows that the implant area is filled with osseous tissue. H&E X20.

left tibia before being applied to the right tibia. observing the deadlines for the rabbit sacrifices. The soft tissue in the left and right tibias was removed by dissection. Slices were taken 5 mm from the side of each implant after the right and left tibias were dissected, the soft tissue was removed to expose the whole bone, and bone blocks containing implants were created. Implant-contained bone blocks were created by cutting slices of bone 5 mm from the sides of the implant. After the soft tissue of the left and right tibias was removed and dissected, the whole bone was seen. Following a 48-hour fixation in 10% formalin, the bone tissue underwent paraffin embedding and alcohol dehydration. The samples were then treated with a formic acid solution to decalcify them. Five-meter slices produced as usual were stained with hematoxylin and eosin. A light microscope was used to evaluate the histology. Histomorphometric measurements were made of the marrow space star volume, trabecular breadth, thread width, cortical bone thickness, and bone cells (osteoblast, osteocyte, and osteoclast).

RESULTS AND DISCUSSION

Two weeks long

A-Group (control)

Histological analysis revealed the formation of osteoid tissue in many areas. (Fig. 1).

B-Experimental group

Under increased magnification, the implant hole area revealed bone trabeculae. In these trabeculae, osteoblasts accumulated around the margins of the vast lacunae filled by osteoocytes.

six weeks duration

A-Control group

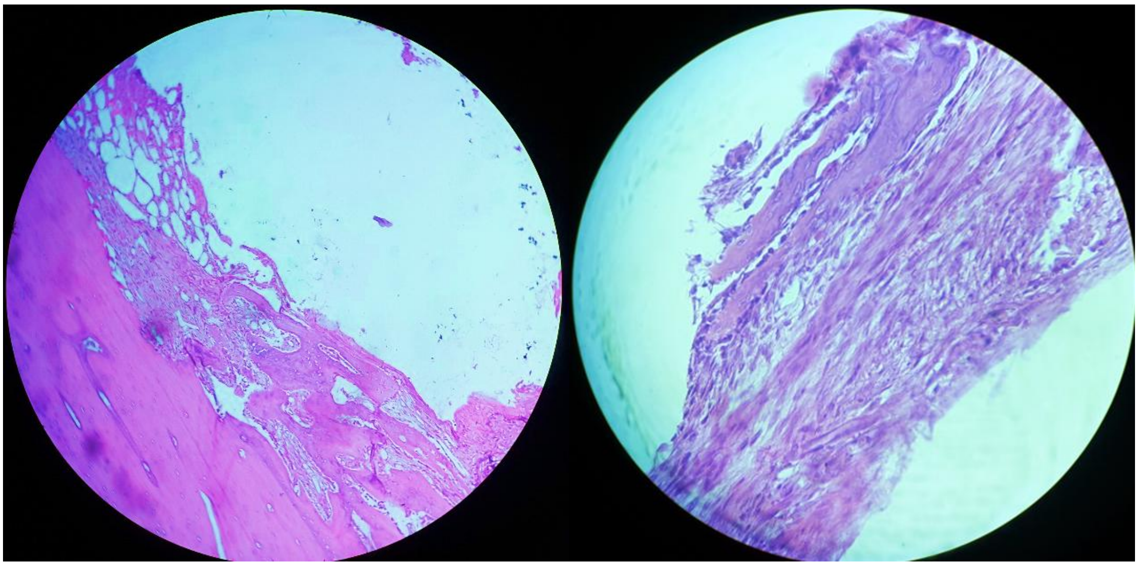
The histology picture clearly shows the development of thick bone trabeculae that are packed with big osteocytes and encircled by osteoblasts. Furthermore, the presence of reversal lines and osteoclasts indicates ongoing bone remodeling (see Fig. 3).

B-Experimental group

The histology image shows mature, well-established bone with osteoblasts surrounding the osteocytes. There are Haversion lamellae in mature bone (Fig. 4).

Histomorphoetric analysis of the examined groups were used to identify the features of the bone architecture

For each healing period, the descriptive statistics for the bone architecture parameters for the experimental and control groups are displayed in (Tables 1 and 2). The mean values of bone formation increase with time for both the



A.X20 **B.X40**

Fig. 2. The six-week experimental group's examination reveals woven bone in the thread area, which is followed by screw-shaped blood vessels and H&E.

experimental and control groups. At every stage of recovery, the titanium oxide and zirconium oxide-coated group's mean values are higher than the control groups. During the healing process, the mean marrow space star volumes in both groups decrease; however, the titanium oxide and zirconium oxide coated group exhibits a more marked decline than the control group. While the mean values for the number of osteoblasts and osteocytes in the titanium oxide and zirconium oxide coated groups increased more quickly at 2 and 6 week intervals than the control groups, both the experimental and control groups' mean values increased over time. Based on the mean results, at the same time in week 6, the titanium oxide and zirconium oxide treated group had fewer osteoclasts than the control group. There was a statistically significant difference between the control and experimental groups in every healing phase, according to an analysis of all

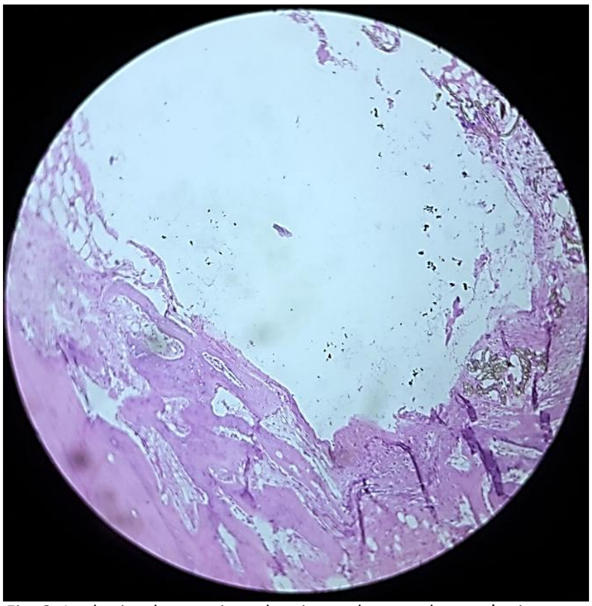


Fig. 3. In the implant region, the six-week control group's view may reveal osteoblasts, osteoclasts (OCL), and osteoocytes (OC). H&E X20.

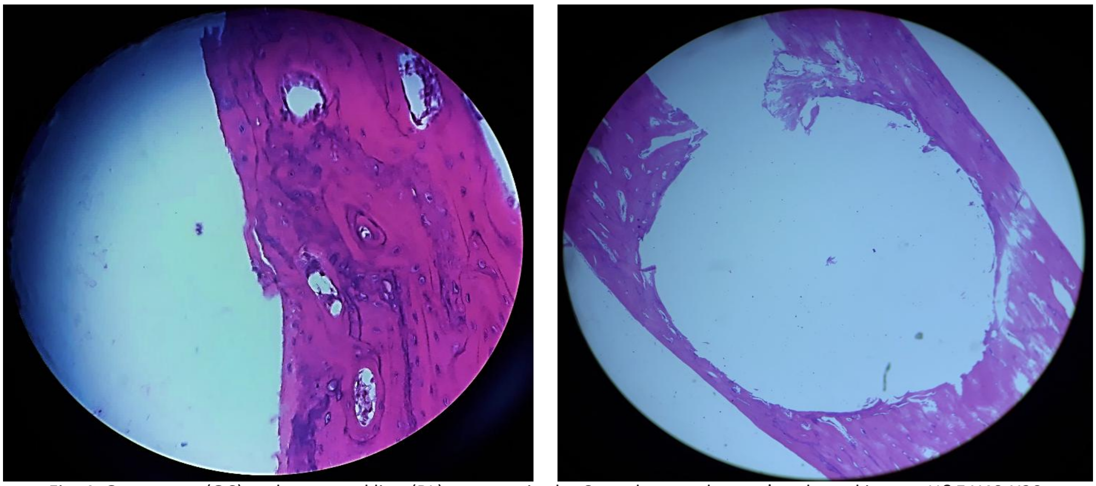


Fig. 4. Osteocytes (OC) and a reversal line (RL) are seen in the 6-week control group's enlarged image. H&E X40-X20.

histomorphometric parameters.

New materials for bone implants are being researched as a result of the rise in orthopedic and dental prosthesis surgery [9,10]. Any implant generally consists of two primary sets of qualities: surface characteristics, which interact with the biologic environment to provide biocompatibility, and bulk material characteristics, which are primarily responsible for mechanical and structural properties [11].

Thin film deposition methods can be used to modify the surface of implanted material. Compared to untreated controls, there was a greater and statistically significant proportion of bone-implant contact in the implants treated with nanoparticles ZrO and TiO_2 [12].

Increased surface roughness, the development of nano-to-microscale porosity, improved hydrophilicity that jointly influenced improved initial clot stability, and increased initial protein absorption—which controls cellular interactions on the implant surface—could all be contributing factors to this. Ca and P ions that may have an impact on early osseointegration were integrated into the oxide layer that was present on the surface. According to Park et al., after six weeks, micro-rough Ti implants in rabbit cancellous bone exhibit considerably improved osteoconductivity thanks to nanoporous oxide layers including Ca and P.These ions' presence, when combined with a porous surface topography, aids in the stability of fibrin, promotes stem cell migration and differentiation, and demonstrates the osteoinductive character of these cells [13–14].

The subsequent deposition rates were acquired for the titanium (Ti) and zirconium (Zr) magnetron sputtering methods in order to attain coating thicknesses between 50 and 100 nm. All areas had a good healing course, according to the histology findings for the experimental and control groups; however, the rates of bone remodeling and deposition changed with each healing interval.

Following a fortnight of implantation in the control animals, the sections manifestly demonstrated the commencement of osteoid tissue development and the replacement of the blood clot by granulation tissue containing a significant quantity of collagen fibers, fibroblasts, and osteoblasts. Osteoblast differentiation had started to replace the granulation tissue in implants treated with ZrO and TiO2 with new bone. [15–16] After a two-week interval, the control group's histology sections showed fragile bone trabeculae with recently formed woven bone. The TiO2 and ZrO treated group with nanotechnology exhibited larger and thicker bone trabeculae in comparison to the control group [17-18].

The results also showed a significant shift in the characteristics of bone architecture throughout time. Increases in trabecular width, cortical width, thread width, and trabecular number over six weeks as opposed to one may be caused by the duration of bone deposition and maturation. On the other hand, quicker bone matrix formation over time may lead to broader bone trabeculae and a decrease in bone marrow star volume. These findings support studies conducted by No additional osteoblasts are required when bone production stabilizes and achieves its maximum size—only what is required to maintain biological activity. This explains why the number of osteoblasts and osteocytes rose with time: every

Table 1. Descriptive data for group's bone architecture characteristics.

Bone Formation	Groups	N	Mean	Std. Deviation	95% Confidence Interval for Mean		Minimum	Maximum
					Lower Bound	Upper Bound		
	control (2w)	16	1.21	0.07	1.03	1.71	1.08	1.42
	control coated (2w)	16	1.55	0.05	1.11	1.9	1.4	1.79

Table 2. Descriptive data for group's bone architecture characteristics.

Bone Formation	Groups	N	Mean	Std. Deviation	95% Confidence Interval for Mean		Minimum	Maximum
					Lower Bound	Upper Bound		
	control (6w)	16	1.76	0.07	1.215	1.46	1.71	1.93
	control coated (6w)	16	2.87	0.1	2.13	2.78	1.99	2.85

new tissue creation requires the generation of additional osteoblasts [19-22].

Additionally, this coating demonstrated improved mesenchymal stem cell adhesion and osteoblast differentiation. When tested in vivo, coated samples outperformed untreated PEEK in terms of bone formation [23,24]. Comparing PEEK implants with and without modifications, those covered with amorphous zirconium oxide with a thickness of nm demonstrate enhanced osseointegration and bioactive characteristics [26-30].

CONCLUSION

In order to optimize the surface characteristics and osseointegration of polyether ketone-based implants for prospective implant applications, thin coatings of nanotitanium and nanozirconium were deposited utilizing magnetron sputtering in this work.

The most crucial elements influencing a bone implant's effectiveness are the osteogenic cells' capacity to make bone, their initial attachment, and their continuing proliferation. Furthermore, the coatings had little effect on the substrates' surface structures or geometry. Regarding their potential uses in the domains of dentistry and orthopedics, the coatings described in this paper show promise.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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