Research Article

Surface Characterization of PEKK Modified by strontium –hydroxyapatite coating as implant material Via the magnetron sputtering Deposition technique

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Abstract Background: The best material for dental implants is polyetherketoneketone (PEKK). However, this substance is neither osteoinductive nor osteoconductive, preventing direct bone apposition. Modifying the PEKK with bioactive elements like strontium hydroxyapatite is one method to overcome this (Sr-HA). Due to the technique's capacity to provide better control over the coating's properties, RF magnetron sputtering has been found to be a particularly useful technique for deposition. Materials and methods : With specific sputtering conditions, the RF magnetron technique was employed to provide a homogeneous and thin coating on Polyetherketoneketone substrates.. the coatings were characterized by Contact angle, adhesion test, X-ray diffraction (XRD), atomic force microscope and Elemental Analysis with Energy Dispersive X-Ray (EDX) Results : indicated that strontium hydroxyapatite had successfully deposited onto the surface with significant improvement in the wettability value to provide a suitable environment for cell attachment, spreading, proliferation, and differentiation Conclusion: Coating PEKK with RF magnetron sputtering can provide homogeneous surfaces laying the groundwork for improving PEKK's potential bioactivity, such as surface wettability. Wetting qualities are critical in implantable materials and are used to predict future osseointegration success.

Keywords: Dental implants, strontium –hydroxyapatite , wettability, Polyetherketoneketone, RF magnetron Sputtering

Introduction

General dentists and a variety of specialists are currently offering implants as a solution for partial and total edentulism. Due to the higher success rate, dental care is turning more and more toward using implant based oral prosthetics. Demand for dental implants has been fueling a rapid market expansion and development of new surgical and prosthodontic techniques implant dentistry continues to evolve and expand [(1)].

Because dental implants come in a variety of materials, dimensions, geometries, surface qualities, and interface geometry, today's dentist must choose from over 2,000 distinct dental implants and abutments for a given treatment setting [(2)].

Mechanizing, electropolishing, plasma spraying, coating, acid etching, surface oxidation, ionization, and phosphate deposit procedures are all used to modify the surface of dental implants. Surface modification of dental implants is thought to be the best way to achieve rapid secondary stability, improve bone-to-implant contact, and shorten the time it takes to restore missing teeth [(3)].

Polyaryletherketones (PAEKs) are a type of high-performance semi-crystalline electronics thermoplastics with a rare combination of thermal resilience, chemical tolerance and outstanding mechanical properties across a wide range of temperatures. This polymer class also possesses higher heat resistance and excellent electrical efficiency[4-6].

In medicine it has been an excellent substitute for titanium in orthopedic applications, and it has been used in dentistry as provisional implant abutment, removable dentures and implanted prostheses and applications continues to grow[7].

Bioactive bone cement, containing strontium hydroxyapatite (Sr-HA), has been designed to have applicable desirable properties [8]. Previous tests found that Sr-HA bone cement did not have cytotoxicity, and was able to support the collapsed spine Sr-HA thin films deposited by pulsed laser deposition on titanium substrates, indicating that the existence of Sr may not only improve the beneficial impact of HA coatings on osteointegration and bone regeneration, but also avoid undesirable bone resorption [9]{Jani, 2015 #621}.

Among the numerous processes, reactive pulsed DC magnetron sputtering surface coating of osseoconductive films has been shown to be successful in increasing the This method is widely used because it produces higher-quality films, has a higher deposition rate, and has excellent long-term process stability. Furthermore, this method can deposit a wide range of compounds, especially thin films on polymers [10].

The quality of the coated film is largely determined by the plasma settings during the sputtering process [11]. The relationship between the features of the deposited film and the chemical compositions of plasma utilized in the sputtering technique should have been used as a criterion for choosing the best plasma conditions for the magnetron sputtering process [12].

In this study, the optimum sputtering conditions for the deposition of Sr-HA film on PEKK using a magnetron sputtering system were accomplished, and the coating layer compositions and roughness were evaluated in relation to sputtering deposition time

Materials and Methods

The pilot study was done for selection the most suitable and appropriates coating technique parameter of the Sr-HA to the PEKK specimens with Magnetron sputtering technique.

Preparation of target

In order to perform the RF plasma sputtering of Sr-HA powder, a circular target of 50mm in diameter and 3 mm in thickness was prepared .The target was made by mixing (30 g) of Sr-HA powder, then loaded and pressed in cylindrical stainless steel mold with dimension of 50 mm in diameter and 3mm in height , the pressing was done under 8 tons pressure for 2 min. using electrical press, to obtain uniform target and to avoid target fracture. The samples were allowed to dry gradually at room temperature for 24 hours to avoid cracks.

RF magnetron sputtering

Radio frequency magnetron sputtering was performed in the system chamber which was made from stainless steel .The target was attached to the anode (positive charge) of system and the substrates were attached to cathode (negative charge) rotating disc.

The PEKK substrates and Sr-HA target were introduced inside the sputtering chamber then the chamber window was closed tightly. The distance between substrates and target was fixed to 10 cm The break-in was performed at a rate of 5 watts (W) per minute up to 150 W operating power, with the source shutters closed, prior to deposition from the HA target. The base pressure was less than $5*10^{-2}$ Pa with an argon gas flow rate of 15 to 20 Sccm and a throw distance of 100 mm. During sputter deposition, the chamber pressure was maintained at 310^{-2} Pa.. The substrates were heated gradually to reach balance temperature of 80° C. The deposition was done at 2 rpm in order to increase the uniformity of distribution. Three sputtering intervals (runs) were performed. The working conditions were summarized in table,[13]

Table 1: The working conditions of deposition Sr-HA on the PEKK substrates

Sample Name	Deposition Time (min)	Pressure (Pa)	Power (W)	Distance between target & substrate (cm)	Substrate Temperature (°C)
Group 1	30	3×10^{-2}	150	10	80
Group 2	60	3×10^{-2}	150	10	80
Group 3	90	3×10^{-2}	150	10	80
Group 4	120	3×10^{-2}	150	10	80

Then coating prepared samples tested adhesion and wettability Thus, the final results determined best condition for coated PEKK by SRHA

Contact angle (Wettability)

Since the wetting properties is very important in implantable material and considered as indicator for future good osseointegration, so surface wettability test (water contact angle test) was used to measure the amount of the PEKK coated layer wettability for the four tested. The disk with low contact angle measurement (high wetting surface) was chosen for PEKK coated with Sr-HA specimens and the screws (experimental group).

Adhesion test

The pull-off test is a more quantitative adhesion test in which an adhesive is used to adhere a loading device, typically known as a dolly or stub, to a coating. A load is gradually given until the dolly is pulled off using a portable pull-off adhesion tester, such as the PosiTest ATM. The tensile strength is measured in pounds per square inch (psi) or mega Pascals, based on the force required to pull the dolly off or the force the dolly withstood (MPa)

A standard method for the application and performance of the pull-off adhesion test is done according to ASTM D4541. Three sample for each groups tested adhesive strength between coating and substrate.

X-ray Diffraction analysis

Phase analysis and structural evaluation was performed for control and experimental groups. Phase analysis was studied using 3121 powders X-ray Diffractometer using Cu Kα target radiation. The 2Θ angles were swept from 20- 60° in step of one degree and Voltage 40.0Kv. The peak indexing was carried out based on the JCPDS (joint committee on powder diffraction standards).

Atomic force microscopical analysis for surface topography:

Atomic Force Microscopy can provide 2D and 3D images of the deposited coat that exhibit average roughness (Ra). This scanning mode was applied to evaluate surface topography of coating. The examined area was 30× 30 μm and 35× 35 μm for the 2 D and 3D micrographs respectively.

In non-contact AFM, a sharp probe is positioned close to the surface under study (order of Angstroms), the probe is then raster scanned across the surface, and the image is built from the force interactions that occur during the scan. A resonator, commonly a silicon cantilever or a quartz crystal resonator, is attached to the probe. Device specifications are DEM (controller: Dual Scope C-21scanner: D3 95-50E. Tip information are (tip curvature<10 nm). Force constant is 42 N/m. Those images had been analyzed using software program to gain the common roughness (Ra), peak-to-valley roughness (Rz) [14].

Elemental Analysis with Energy Dispersive X-Ray (EDX)

It is an x-ray technique which is also referred to as (EDX) (energy dispersive X-ray analysis) or (EDS) (energy dispersive spectroscopic) used to identify the materials elemental composition, mapping of the elemental of the analyzed sample and image analysis.

The main principle of spectroscopy is that each element has a unique atomic structure, allowing for a unique set of peaks on its electromagnetic emission spectrum. For elemental analysis or chemical characterization of a sample, it relies on the interaction of some source of X-ray excitation and a specimen. The consequence of exposing a sample to a high-intensity x-ray is the distinctive X-ray of the elements included in the sample, which was identified by the detector. The detector's signal was decoded and evaluated [15, 16].

Earlier than detection, samples were sputtered with a skinny layer of platinum (white gold) using a sputtering device to enhance surface conductivity. [17].

Results

Wettability (Contact angle)

The result of measuring of the water contact angle for the samples was showed that the contact angle for the control PEKK was (81.14) , 30 min. (2.49), 60 min. (2.19) , 90 min. (1.02), and decrease to 0.28 in the 120 min. group, each of these results was repeated three times for each sample and the number above was the average for the readings group.

Water contact angle images were taken for all study groups (Descriptive statistics) were summarized in table (2).

Statistically F-test of one way ANOVA test shown a highly significance difference in the water contact angle among the five groups, P≤ .01 at three degrees of freedoms as shown in table (3).

Table 2: Descriptive statistic of water contact angle test

test	**groups**	**Mean**	**SD**	**SE**	**Maximum**	**Minimum**
water contact angle test	**control**	81.1433	1.72639	.99673	82.16	79.15
	30 min.	2.4910	.79450	.45870	3.39	1.90
	60 min.	2.1973	.53113	.30665	2.65	1.61
	90 min.	1.0260	.25436	.14686	1.30	.80
	120 min.	.2863	.40127	.23168	.75	.01

Table 3: ANOVA test of water contact angle test

tests	**Within Groups**			**Between Groups**			**F**	**Sig.**
	Sum of Squares	**df**	**Mean Square**	**Sum of Squares**	**df**	**Mean Square**		
water contact angle test.	8.239	10	.824	15232.780	4	3808.195	4622.171	0.000 [HS]

the results of the bond strength testing for each coating type. The maximum adhesion strength of SR HA layer deposited on a PEKK was 2.59 MPa. (standard range 0.4 -3.3 MPa) was found 120 min. deposition time for SR HA coatings group.

Table 4 shows the stud pull bond strength of SR HA coatings on PEEK substrates from this study compared. (Descriptive statistic of adhesion test)

test	groups	Mean	SD	SE	Maximum	Minimum
adhesion test	30 min.	.620	.1389	.0802	.78	.53
	60 min.	.880	.2787	.1609	1.20	.69
	90 min.	1.290	.5602	.3234	1.86	.74
	120 min.	2.590	.7617	.4398	3.18	1.73

Table 5: ANOVA test of adhesion test

tests	Within Groups			Between Groups				
	Sum of Squares	df	Mean Square	Sum of Squares	df	Mean Square	F	Sig.
adhesion test.	1.982	8	.248	6.885	3	2.295	9.261	.006

Topographical analysis (AFM)

Surface roughness analysis interpreted by atomic force microscope for both PEKK and PEKK coating with SR HA specimen surface is shown in figure (1) to identify number of morphological changes on the surface which is roughness value (Ra) measured by this technique is about 35.1 nm. and increase to 43.2 nm when coating with SR HA

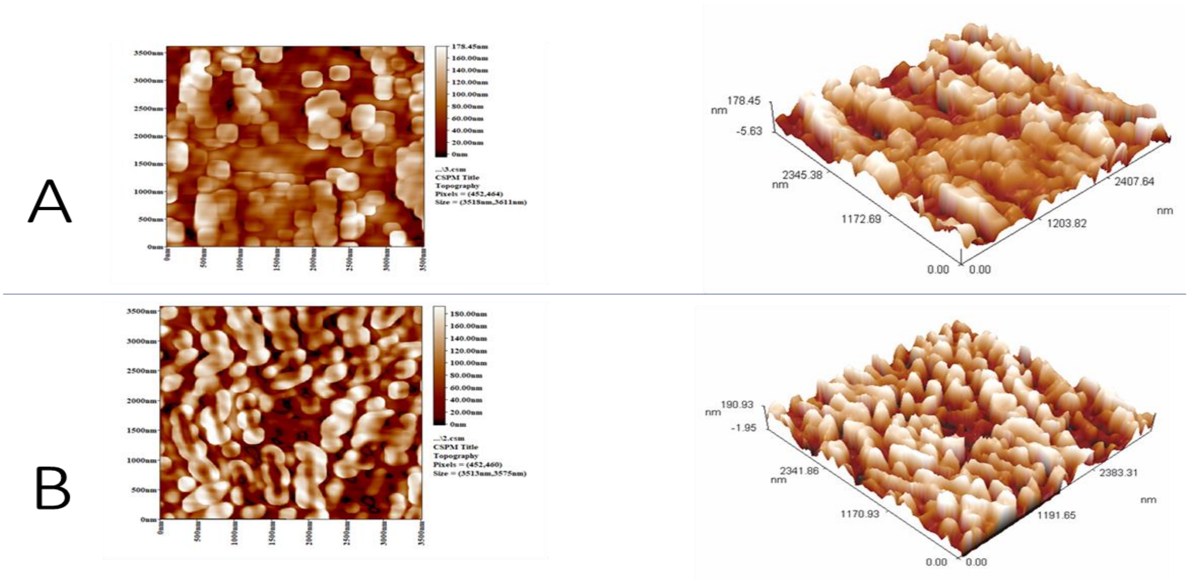

Figure 1 : AFM micrographs, 3 D and 2D graphs for the average roughness of specimen.

A. AFM micrographs for PEKK, B. AFM micrographs for PEKK coating with SR HA specime

Xray diffraction

The phase analysis was carried out on 3121 powders using a Cu K radiation X-ray diffractometer. The 2θ angles were swept in one degree increments from 20 to 60 degrees. The peak indexing was done using the International Centre for Diffraction Data's JCPDS (joint committee on powder diffraction standards), ICDD file #25-0891 ,#33-1322 , # 9-432 for Sr-HA and show the typical XRD patterns of the Sr-HA coating on pekk implants.

The PEKK showing diffraction peaks at 18.7°, 20.8 °, 22.9° and 28.9°, and these corresponded to the diffraction planes of (110), (111), (200) and (211) as based on the previous literature [18]

The strongest line of this phase are (602),(332),(105),(333),(615) and (722) at 2Θ 22.6495 , 25.4855 , 34.1999 , 39.4931 , 40.3676 and 41.2593 respectively.

The presence of PEKK peaks in the XRD pattern after coating process is due to the penetration of X rays beyond the coated layer

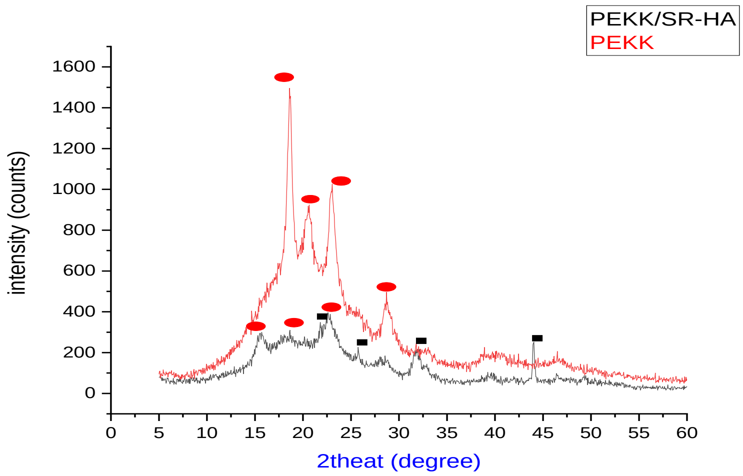

Figure (2) : XRD pattern of PEKK and PEKK coating with Sr-HA specimen

An EDX spectrum manifestation the atom peaks correlate with the greatest X-rays received energy levels of PEKK and PEKK coating with SR HA; the greater peak in a spectrum, the more element concentrated, so the greatest level can be as seen in fig (3).

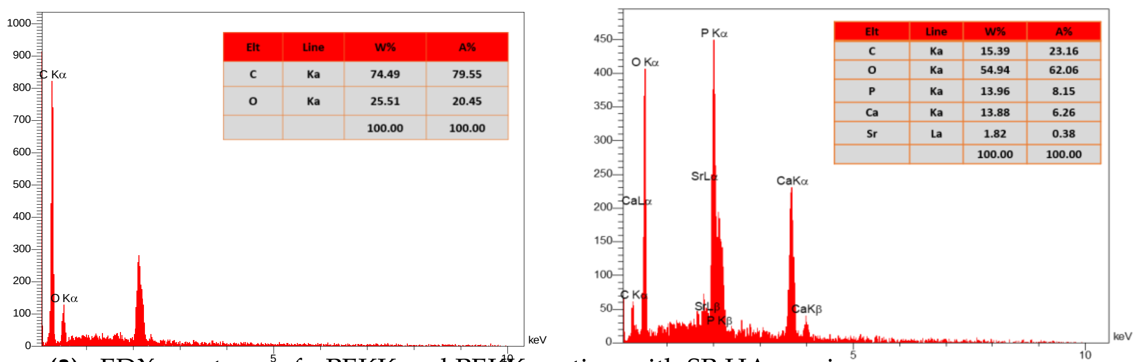

Figure (3) : EDX spectrum of PEKK and PEKK coating with SR HA specimen

Discussion

Radiofrequency (RF) magnetron sputtering as an alternate method for covering implants with ceramic coatings. We recently reported on RF sputtering as a viable method for covering a substrate with a biocompatible ceramic layer.

The advantages of RF sputter-coating over conventional techniques, based on these preliminary findings, are superior adhesion, thickness homogeneity, and the capacity to coat implants with complex surface geometries.

Since the wetting properties is very important in implantable material and considered as indicator for future good osseointegration, so surface wettability test (water contact angle test) was used to measure the amount of the PEKK coated with Sr-HA layer wettability for the ten tested for different sputtering time (30, 60, 90 and 120 minutes)

The disk with low contact angle measurement (high wetting surface) was chosen for Sr-HA coating PEKK as the final experimental specimens.

Regarding the result of measuring of the water contact angle for the samples is showed that the contact angle for the control PEKK was (81.14) , 30 min. (2.49), 60 min. (2.19) , 90 min. (1.02), and decrease to 0.28 in the 120 min., the decrease in the contact angle value could be attributed or explained due to PEKK is hydrophobic and bio-inert, which has limited its broad application [19, 20], its hydrophilic due to its hydroxyl groups and hydrophobic aromatic ring, while HA is hydrophilic due to its hydroxyl groups [21]. A HA coating has been used in several research to transform hydrophobic substrates to hydrophilic surface materials [22]. Several studies have employed a HA coating to convert hydrophobic substrate to hydrophilic surface material Furthermore, HA has the ability to recruit osteoblasts as well as enhance cell proliferation and osteogenic differentiation in osteoblast cells [23, 24]. In this study, HA was used to modify the surface of PEKK, resulting in a hydrophilic and bioactive surface that was ideal for cell adhesion, spreading, proliferation, and differentiation [25].

Coatings must adhere to the substrates they are applied to in order to perform successfully. To determine how well a coating is bound to the substrate, a variety of well-known procedures can be used. A pull-off adhesion tester is commonly used to perform common measuring techniques.

The most significant aspect that defines the quality of the applied coating and its clinical application is the adhesive strength between Hap coating and substrate [26]. Surface chemistry, microstructure, surface roughness of the substrate, and processing parameters of the applied technique all influence the bonding strength of hydroxyapatite and substrate. In general, mechanical interlocking and chemical bonding can cause adhesion between coating and substrate, which is consistent with the findings of this study, which show that adhesion increases with deposition time.

Also the biocomposite deposited film by RF magnetron sputtering under specific working parameters showed a higher roughness surface which consequently enhance bone growth and biological fixation , this finding is in agreement with (Pietro *et al.*,2016)[27] and (Prosolovab *et al.*, 2017)[28] who proved that films precipitated by plasma sputtering are characterized by increased roughness of substrates, which can be seen in AFM result as surface roughness increase with coating layer produce by RF magnetron sputtering as Ra (roughness average) for coating sample is (43.2nm) and for control group (35.1nm). At specified working pressure ($3x\ 10^{-2}$) , sputtered atoms or clusters reached the substrate with reduced kinetic energies , which caused the surface roughness to be increased [29]. This can be explain increase wettability of PEKK coated with Sr-HA than uncoated, rough surface is a crucial property must be controlled in processing of implant due to it has superior adsorption of biomolecules from surrounding fluids, in addition affecting in stress distribution at the implant bone line contact, the surface roughness in nano level may be increase cell growth and osteoblastic differentiation, also inhibit colonization of bacteria lead to healing developing with short time before loading [30], as explained by (Deng et al., 2015),[31] this is agree with *Rong et al., 2009; Eom et al., 2012* [32, 33] who seen that the surface roughness was significantly increase when coated by material with nano or micro particle size.

The EDX analysis of the of PEKK coated group shown that, calcium, phosphate, strontium , oxygen and carbon were the chief elements in their surface that indicated the successful coating Sr-HA on PEKK substrate. In group P, the oxygen and carbon elements referred to PEKK material, while the calcium, strontium element referred to Sr-HA. This findings are confirmed the formation of SR HA coating layer showed by XRD.

The presence of strontium and hydroxyapatite was demonstrated by the X-ray diffraction pattern of a coated layer of strontium-hydroxyapatite. XRD revealed that narrow peaks indicated a layer with a high level of crystallinity, whereas broad peaks indicated a layer with a lower level of crystallinity [(34)]. The amount of strontium-hydroxyapatite element in EDS can also be noticed. PEKK peaks appear in the XRD pattern following the coating procedure because X rays penetrate beyond the coated layer.

Conclusion

Coating PEKK with RF magnetron sputtering can provide homogeneous surfaces laying the groundwork for improving PEKK's potential bioactivity, such as surface wettability. Wetting qualities are critical in implantable materials and are used to predict future osseointegration success.

Conflict of interest: None.

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التوصيف السطحي لبولي إيثير كيتون كيتون المعدل بواسطة طلاء السترونشيوم-هيدروكسيباتيت كمادة غرسية عبر تقنية ترسيب الاخرق المغنطروني

الباحثون: غسق هشام جاني[1] , عبد الباسط احمد

المستخلص

فضل مادة لزراعة الأسنان هي مادة البولي إيثير كيتون كيتون. ومع ذلك ، فإن هذه المادة ليست حاثية عظمية ولا موصلة للعظم ، مما يمنع توضيع العظام المباشر. تعديل البولي إيثير كيتون كيتون. مع العناصر النشطة بيولوجيًا مثل السترونتيوم هيدروكسيباتيت هي إحدى الطرق للتغلب على هذا السترونشيوم هيدروكسيباتيت. نظرًا لقدرة التقنية على توفير تحكم أفضل في خصائص الطلاء ، تم العثور على رش المغنطرون ليكون أسلوبًا مفيدًا بشكل خاص للترسيب.

المواد والطرق: في ظروف رشاش محددة ، تم استخدام تقنية المغنطرون لتوفير طلاء رقيق متجانس ومتجانسة على ركائز بولي إثير كيتون كيتون .. تميزت الطلاءات بزاوية التلامس واختبار الالتصاق وانحراف الأشعة السينية ومجهر القوة الذرية والتحليل العنصري مع الأشعة السينية المشتتة للطاقة

النتائج: أشارت إلى أن السترونتيوم هيدروكسيباتيت قد ترسب بنجاح على السطح مع تحسن كبير في قيمة قابلية البلل لتوفير بيئة مناسبة لربط الخلايا وانتشارها وتكاثرها وتمايزها.

الخلاصة: طلاء بولي إيثير كيتون كيتون. مع رش المغنطرون يمكن أن يوفر أسطحًا متجانسة تضع الأساس لتحسين النشاط الحيوي المحتمل لـ البولي إيثير كيتون كيتون ، مثل قابلية السطح للبلل. تعتبر صفات الترطيب حاسمة في المواد القابلة للزرع وتُستخدم للتنبؤ بنجاح الزراعة العظمي في المستقبل.