

# Implant Surface Modifications: A Review

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## ABSTRACT

The aim of the present review was to elaborate on the surface modifications of biomaterials which are used in implant dentistry. The ongoing studies on the clinical and laboratory phases and on the biomaterial sciences have largely aimed at invoking a stronger bone response and an earlier and longer functional loading. Surgical grafting procedures to improve the bone bed are used to create an ideal environment for the implant functioning. The implant selection which is based on the available bone, is also an important determiner of the long term clinical success. The interfacial zone between the implant and the bone is composed of a relatively thin layer (<100µm) which consists of heterogenous metallic oxide, proteins and connective tissue. The integrity of the implant – tissue interface is dependent on the material and on mechanical, chemical, surface, biological and local environmental factors, all of which change as a function of time in vivo.

Because a stable interface must be developed before the loading, it is desirable that the tissue apposition may be accelerated to the implant surface. The material developments that have been implemented in the clinical practice include the use of surface roughened implants and bioactive ceramic coatings. Osseointegration occurs around the screw threaded implants through the tissue ongrowth or through a direct apposition between the tissue and the implant surface. The alternative methods of the implant-tissue attachments, based on the tissue ingrowth into roughened or three dimensional surface layers, yield a higher bone metal shear strength and decreased implant loosening. A positive effect of various surface modifications which are illustrated in this review, has been observed and suggested by many groups.

**Key Words:** Hydrophilic surface Laser etching Nanotitania Osseointegration Anodized implants Biotolerant

## INTRODUCTION

An implant is a medical device which is made from one or more biomaterials, that is intentionally placed in the body either totally or that is partially buried beneath an epithelial surface [1]. Osseointegration is the foundation of implant sciences and infinite articles have been published on the various aspects of manufacturing the implants and on the clinical and laboratory phases of implants. The implant machining, surface, designing, surgical techniques and the peri-implant considerations have all progressed from infancy to the state of art and science and continue to evolve with each passing year. The surface characteristics at the micro or nanometre level, hydrophilicity, biochemical bonding and other features are few of the determiners which are responsible for the implant's success [2].

Osseointegration per se is not linked to certain defined surface characteristics, since a great number of different surfaces achieve osseointegration. However, the stronger or weaker bone responses may be related to the surface phenomenon [2]. The bone implant interface can be controlled by the selection and modification of the biomaterial from which is made. These include morphological, physiochemical and biochemical methods. The morphological methods involve alterations in the surface morphology and roughness, such as hydroxyapatite coating or blasting and etching. The physiochemical methods involve modification of the surface energy, the surface charge and the surface composition. The biochemical surface modification endeavours to utilize the current understanding of the biology and the biochemistry of the cellular function and differentiation [3]. The biomaterials which are used

most commonly for the dental implants are metals and their alloys, namely commercially pure titanium (1-4grades) and titanium alloys like Ti-6Al-4V, which are used most commonly as endosseous implants. The metallic implants undergo modifications such as passivation, anodization, ion implantation and texturing [4]. Our aim was to review the surface modifications of the titanium based implants, some of the techniques of the modifications and the newer formulations.

## MORPHOLOGICAL SURFACE MODIFICATIONS

### A) Machined and Blasted Surface

Alterations in the biomaterial surface morphology have been used to influence the cell and tissue responses to the implants. Porous coatings were originally developed with the consideration of the pore size (50-400µm optimal) and the volume fraction porosity (35-40% optimal) [3]. In initial studies, screw shaped implants were prepared with different surface topographies as machined and blasted surfaces and the topography was measured by using a confocal laser scanning profilometer, their surface roughness being characterized by using height and spatial descriptive patterns [5]. In implant research, the term 'machined surface' is often used as a description of a turned, milled or sometimes a polished surface [6]. The turned surface has an average roughness of 0.96µm and an average peak spacing of 8.6µm. Blasting the implant screws with 25-75µm alumina particles results in an isotropic surface with average height deviations of 1.1 and 1.5µm respectively. Blasting with 250µm alumina particles results in a less isotropic surface and

an average height deviation of about 2.0 $\mu\text{m}$  [5]. Several commercial implant systems are machined with the turning process and the surface roughness which is achieved with the 250 $\mu\text{m}$  blast particles is comparable to that of some commercial plasma sprayed and hydroxyapatite coated implants [7]. A series of investigations have demonstrated a firmer bone fixation of the implant with an average surface roughness (sa) of 1-1.5 $\mu\text{m}$  than those of smoother implants with an average surface roughness of 0.6 $\mu\text{m}$ . A comparison between the 25 and 250 $\mu\text{m}$  blasted specimens (by using Basic Due Equipment Renfert, Gottingen, Germany with a speed of 3 seconds per revolutions and a pressure of 0.36MPa) showed significantly more bone to be in contact with the implant surface for a smoother 25 $\mu\text{m}$  blasted specimen after 4 weeks in rabbit bone, while the same comparison after 12 weeks revealed no difference. A better bone response was seen at surfaces with an intermediate roughness of 1.5 $\mu\text{m}$  and average height deviation than with either a smoother (sa 0.5 $\mu\text{m}$ ) or a rougher (2.0 $\mu\text{m}$ ) surface. However, the orientation of the surface structure is also an important factor for the bone tissue implants, as has been already concluded for soft tissue implants [8]. The enlarged surface topography seems to enhance the bone fixation after one year of follow up [5]. Morphometric [9] analyses have shown a difference in the bone implant contact percentage with the varying of the surface characteristics as well as the sensitivity of the cells to the surface microtopography. The surface modifications, through a variety of processes, have resulted in an increased bone to implant contact and biomechanical fixation at the earlier implantation times as compared to the machined implants [10].

A positive correlation has been found between the increasing surface area and the increasing ion release [11,12]. In the past, some studies had expressed concerns about the leakage of different metallic ions from the implants to the hard and soft tissues and their potential negative effects on the host [13,14]. The primary interaction between the implant material and the host takes place at the surface in the region of the approximate size of one water molecule (~0.1-1.0nm) and high levels of Ti in the spleen and lungs of rabbit have been reported immediately after surgery, but these were well within the normal limits. Humans normally have Ti levels of 50 ppm, but these can reach upto a level of 300ppm in the surrounding tissues. Kasemo and Lausmaa (1991) demonstrated the dissolution of the corrosion products into the biofluid and the adjacent tissues [4]. Upto 1,600 ppm of titanium ions have been found in the haversian canals in relation to the titanium implant [15]. Ducheynes concluded that the titanium ions stay in the vicinity of the implants because of their acidic properties and their limited coordinated capability to the representative cellular chelators in physiological conditions [16]. The released ions could have a local negative effect and Blumenthal and Cosma [17] found that the released titanium and vanadium ions interfered with the mineralization of the bone which was adjacent to the implant surface. Thus, an enlarged surface area may have a negative effect as a result of the enhanced ion release [5]. Other investigators did not find any systemic or local toxicities which were caused by the ions which were released from the implants [18,19].

## B) Blasting and Acid Etching (Physiochemical Methods)

Sand and grit blasting are used to modify the implant surface by using titanium oxide and alumina particles. 25 $\mu\text{m}$  particles of TiO<sub>2</sub> are used to grit the blast. The large grit sandblasting particles are corundum 0.25-0.5mm and the medium grit particles are

250-500 $\mu\text{m}$  in size. Acid etching can be done by using an HCL/H<sub>2</sub>SO<sub>4</sub> mixture or by pickling in 2% HF/10%HNO<sub>3</sub>. These processes leave pits and craters. In addition to the surface roughness, sand blasting and acid etching can remove the surface contaminants and increase the surface reactivity of the metal [9].

Sand blasted and acid etched surfaces have a hydrophobic surface and the new SLA active implants have a hydrophilic surface which shows a stronger bone response. These have an sa of 1.75 $\mu\text{m}$  and a developed surface area ratio (sdr) of 143%, which is indicative of the high density of the peaks than are seen in the SLA implants [2]. The original Branemark turned pure Ti implants had an sa of 0.9 $\mu\text{m}$  and a developed surface area ratio (sdr) of 34%. The different etching processes also may lead to the formation of Titanium hydrides (TiH<sub>2</sub>, TiH<sub>3</sub>, TiH<sub>4</sub> or a combination which needs investigation) and the replacement of hydride by oxygen results in the slow transformation of the implant surface, resulting in nanometre sized particles of titanium on the surface. The nano roughness may be important in the protein adhesion, immediately after the implant placement [2]. Sand blasting and etching can increase the rate and amount of the bone formation. [9] The alkaline phosphatase specific activity was enhanced and osteocalcin production, the latent transforming growth factor beta and prostaglandin E2, all which were involved in the bone formation were found to be increased [9].

## C) Fluoride Surface Treatments

Jimbo et al [20] showed the surface hydrophilicity of the implants when they were treated with fluoride containing acids [21]. Based on biomechanical and histomorphometric data, the fluoride modified titanium implants demonstrated a firmer bone anchorage than the unmodified implants, after a short healing period. The formation of fluoridated HA and fluorapatite in the calcified tissues has been demonstrated. The increased seeding rate of the apatite crystals, the stimulation of the osteoprogenitor cells, an increased alkaline phosphatase activity and the incorporation of newly formed collagen into the bone matrix are the reported effects of the fluoride modification.

In one study [22], the chemically modified sand blasted and acid etched surfaces showed a successful functional loading with the hydrophilic implants as early as 3 weeks in the maxillary molars by using the cited protocol.

## D) Anodized Surface Implants

Anodized surface implants [2] are implants which are placed as anodes in galvanic cells, with phosphoric acid as the electrolyte and current is passed through them. The surface oxides grow from the native state of 5nm to approximately 10,000nm. The sa of TiUnite is reported to be 1.1 $\mu\text{m}$  and its sdr 37%. [2] Another study [23] reported the thickest Ti Unite surface oxide coating of 2 $\mu\text{m}$  with anodic oxidation, with the implant having a rough surface with a pore size distribution of 0.06-12 $\mu\text{m}$ , showing the presence of micro and nano pores. According to Hall and Lausmaa, there is 5% phosphorus in the surface layer in the form of phosphates. At the implant surface, there is amorphous TiO<sub>2</sub> and the crystalline grains which are present in the amorphous matrix are of anatase TiO<sub>2</sub>, although few spots could originate from thermodynamically more stable rutile. Anodic oxidation results in the growth of a native titanium oxide layer and a porous topography, with the bone formation occurring directly on the moderately rough oxidized surface [23].

## E) Laser etching and Micro Arc Oxidation

Other methods of surface modification are laser etching and micro-arc oxidation [24]. The implants which are modified by micro-arc oxidation show an increased bone response as an increased cell contact, spread and removal of the torque as compared to the turned implants [25]. The laser method is a process which can be used to produce an implant surface with enough roughness for good osseointegration. Laser treated Ti implants had reduced surface contamination [26] and experimental studies showed increased removal of the torque for the laser etched implants which were placed in rabbit tibia, in comparison to the turned surfaces [27,28].

Laser etching and micro-arc oxidation were both used for the implant surface modification in this study [24]. Laser etching may be a promising new method which can be used to pretreat an implant surface prior to MAO. After the implants are ultrasonically cleaned, they are laser etched by using an Nd:YAG laser at a power of 50kw, frequency of 7.5khz and 16.4A current. They are then processed in an electrolyte solution with 3.5% glycerophosphate disodium salt pentahydrate and 1.2% calcium acetate monohydrate by micro-arc oxidation (voltage 350v, frequency 800hz) for 15 seconds. X-ray diffraction analysis showed an anatase titanium oxide phase. The surface element composition which was identified by an electron microscope revealed Ti, C, O, Ca and phosphonium. Laser treatment improved the bone response and ideal pores with a specific diameter, depth and intervals can be controlled. Itala et al observed that the optimal pore size which was needed to encourage the mineralized bone was 100-400µm [29]. Micro-arc oxidation produces a titania film with a porous structure and micropores of 1-5µm. Ultrastructural changes in the oxide layer were found to be closely related to the voltage which was used for the micro arc oxidation. An increased voltage led to an increased roughness, pore size and thickness of the oxide layer [30]. At 350 V, only TiO<sub>2</sub> in the anatase phase exists and with an increased voltage, rutile TiO<sub>2</sub> is formed and it increases along with anatase [31]. The corrosion resistance of the TiO<sub>2</sub> layer on the MAO implants is improved three times the value for Ti and two times the value for the dense layer or the layer which is formed when a pure Ti surface is exposed to air and oxidized [32]. The production of prostaglandin-2 and transforming growth factor beta-1 is enhanced by the surface roughness, thus suggesting that the surface roughness can mediate the autocrine and paracrine regulation of the osteogenesis and also modulate the effects of the systemic hormone, 1,25 dihydrovitamin D3 on the osteoblasts [33]. Laser treatment leads to contamination with carbon and oxygen, with 1.44% carbon on the surface. Deppe et al [34] determined that carbon dioxide from the air may have provided the carbon and that laser was considered to be least contaminating surface treatment in comparison to the acid etching, sand blasting or the plasma spraying technique. This study also found that the MAO Ti implants that included calcium and phosphorous had the capability to induce the formation of bone like apatite in the simulated body fluids [35]. The mechanism of osseointegration of the oxidized implants has been shown to be mechanical interlocking and biochemical bonding [36].

## SURFACE COATINGS

### Ceramics

Bioceramics can be categorized as bioactive (bioglass/glass ceramic), bioresorbable (calcium phosphate) or bioinert (alumina,

zirconia and carbon). Dressman first reported the use of ceramic (plaster of paris) to repair bone defects in the 1980's. The interest in the invention of bioceramics was developed in 1960 by the works of Hulbert and co-workers. The modern era can be traced by Smith's study of the ceramic bone substitute, Cerosium (1963) which was composed of porous aluminate ceramic which was impregnated with epoxy resin. The first commercially available HA coated implants were seen in the 1980 s [1]. Alumina implants in polycrystalline forms (Frialit-1) or as a single crystal (Kyocera) were tried clinically more than 30 years ago. However, the material did not survive the scrutiny of time and it was withdrawn [2]. Then followed implants which were coated with HA, of which the 1st generation did not work well. The 2nd generation HA coated implants demonstrated acceptable 5-year results in at least one study. HA forms a strong chemical bond with bone due to the presence of free calcium and phosphate compounds at the implant surface. The HA ceramic is the only material in which all the osteotropic phenomena (epitaxy, apatite protein affinity and structural osteotropism) are combined. The novel modes of application of HA resulted in much thinner HA layers than those which were used previously, when plasma was sprayed with a minimum coating of 50µm thickness. The modern HA applications of 1µm or nanometre thickness have reduced the risk of loosening of the implant from the surface. [2] Hydroxyapatite or tribasic calcium phosphate has largely eclipsed tricalcium phosphate coated implants [3]. The HA coating consists of amorphous and crystalline forms with a large density of cracks. An HA coated implant surface roughness, sa of 3.29± 1.5µm has been reported [23], with the top 1-2 µm of the HA layer being amorphous, while the rest of the crystalline layer was hexagonally packed. Crystalline coatings are superior to the amorphous and uncoated implants with respect to the bone implant contact. One study showed that the low crystallinity plasma sprayed implants (46%HA) exhibited three times dissolution of the calcium ions as a higher crystallinity (75% HA) material.

### Preparation of dense ceramic implants by compression and subsequent sintering [3] :

(a) **Powder precompressed** in Perspex die by means of an upper and lower punch. To prevent the powder from sticking to the inner surface of the die, stearic acid in alcohol is applied as a lubricant. After the powder compact is pushed out, it is placed in a rubber tube, brought under vacuum and is isostatically compressed (100MN/m<sup>2</sup>) in an oil containing pressure vessel (density 44%). It is heated at a temperature increase of 100°C /hr in a wet oxygen atmosphere for 6 hours and is cooled over slowly at 100°C/hr.

(b) **Hot isostatic pressing** – is used to develop the highest density and the strength which are possible in crystalline ceramic material. Both heat and pressure are used to enhance the ceramic density. This technique was extensively described by Oudemans (1969). Sintering occurred at 900°C and the pressure which was applied on the upper punch was 50MN/m<sup>2</sup>, with the pressing rate optimal at 25mm/hr.

### HA coating on the implant surface (50-70µm) by various methods:

1) **Plasma spraying** – The stream of the HA powder is blown through a very high temperature flame that partially melts and ionizes the powder, which emerges from the flame, hitting the metallic surface which has to be coated. This method uses carrier gas which ionizes the forming plasma and superheats the particles of HA, which undergo partial melting and are propelled towards the surface which has to be coated, producing around 50µm

thick coatings. The most stable of the plasma sprayed calcium phosphate coatings is fluorapatite. The plasma sprayed HA coatings rely on the mechanical interlocking between a grit blasted or etched metallic surface and ceramic for physical integrity during the implant placement. This specific interface between the bulk metal, the metal oxide and the bioceramic coating has been regarded by some as weak link, where adhesive failures during insertion or after osseointegration have been reported [10].

2) **The vacuum deposition techniques** involve the bombardment of a target in a vacuum chamber, resulting in sputtered or ablated atoms being coated on the positioned substrate. These techniques include ion beam sputtering, radiofrequency sputtering (a radiofrequency magnetic sputtering apparatus with a base pressure of  $10^{-6}$  mbs ; the sputtering is performed in a mix of argon and reactive gases to obtain a desired HA stoichiometry [37]) and pulsed laser deposition.

3) **The sol gel and dip coating method** – In this technique, the coating is fired at 800-900°C to melt the carrier glass to achieve its bonding to the metallic substrate. The precursor of the final product is placed in the solution and the metal implant which has to be coated is dipped into the solution and is withdrawn at a prescribed rate. It is then heated to create a more dense coating.

4) **Electrolytic process** – Electrophoresis and electrolytic deposition are two processes that deposit HA out of a bath of proper chemistry. The porous surface materials can be uniformly coated and the original composition of the ceramic can be maintained [3].

The modern HA [2] applications may be of 1µm or of even nanometer thickness and therefore, the risk factor can be assumed to be lower, should HA loosen from the substrate. The Nanotite implant has a 20 nm HA compound attached to its surface. The HA nanoparticles [38] were prepared by mixing phosphoric acid and calcium nitrate with a Ca/P molar ratio of 1.67 in the presence of a liquid crystalline phase which was built of surfactants, water and water insoluble organic solvents. The liquid crystalline phase works as a template, hindering the particle growth, limiting the particle size to 5nm. After the particles were formed, the liquid crystalline phase was dissolved and the particles were deposited onto Ti implants by a dip coating technique and were dried. The surfactants which were absorbed during coating process were burned away at 550°C for 5 minutes in an atmosphere of nitrogen. This resulted in the nanocrystalline HA thin layer being deposited on the implant surface. Micro-arc oxidation also produces a titania film with porous structures of 1-5µm. The existence of TiO<sub>2</sub> on the Ti surface has been reported to improve the bone formation. Topographical evaluations suggested decreasing surface roughness from polished>nano HA>nanotitania.

The Zirconia ceramic implants are another example of implant ceramics which were first successfully used in the 1960s. As compared to alumina, the partially stabilized zirconia (Yttrium stabilized tetragonal polycrystalline Zr) has higher flexural strength and fracture toughness, a high Weibull modulus and a lower Young's modulus, with an ability to be polished to a superior surface finish [1]. Degradation free new TZP/alumina composites and some with 0.05mol% TiO<sub>2</sub> are being investigated. The study of Gahlert et al demonstrated a stronger bone response to the sand blasted / acid etched zirconia implants surface (sa 1.15µm), followed by the rougher of the two (sa 0.13µm-0.56µm), the ZrO<sub>2</sub> implant. Wennerberg/ Albrektsson recommended proceeding with

caution and slowly with the zirconia ceramic coated implants [2].

### Nanotitania coatings

Nanotitania coatings were prepared during a study [38] by using the sol-gel technique. Commercially available tetra isopropyl orthotitanate was dissolved in absolute ethanol. Ethyleneglycol monoethylether, deionized water and fuming HCl 37% were dissolved in ethanol. The two solutions were mixed rapidly and stirred effectively for 3 minutes. The coating sol was aged at 0°C for 24hours before the Ti substrates were dip coated and the substrate was withdrawn at 0.30mm/s. The coated substrates were heat treated at 500°C for 10minutes, cleaned ultrasonically in acetone for 5 minutes and dried at ambient temperature [38]. The Nanotitania implants had an increased feature density and a large feature coverage area as compared to the nano-HA implants. This could present more binding sites for the protein cell attachment and for increased bone contact. The Nanotitania implants exhibited an ordered arrangement, forming a homogenous layer on underlying topography. The Nano-HA implants revealed nano HA features in being placed in a semioordered arrangement and not covering the entire surface.

**New surfaces** – Silicon substitution [1] has led to the development of macroporous silicon substituted hydroxyapatite implants. New substitution with the carbonate or the yttrium ions and different levels of substitution may lead to further improvement in the bioactivity of HA.

### Biochemical Methods of the Surface Modifications

The biochemical methods of the surface modifications offer an adjunct to the physiochemical and the morphological methods. Their goal is to immobilize proteins, enzymes or peptides on biomaterials for the purpose of inducing specific cell and tissue responses, or in other words, to control the tissue-implant interface with molecules which are delivered directly to the interface. One approach uses cell-adhesion molecules like fibronectin, vitronectin, TypeI collagen, osteogenin and bone sialoprotein. The second approach uses biomolecules with osteotropic effects which range from mitogenicity (interleukin growth factor-1, FGF-2, platelet derived growth factor –BB) to the increasing activity of the bone cells, which enhances the collagen synthesis for osteoinduction [3].

The application of various biotolerant agents, for example, rhBMP-2, within the confined boundaries of the hollow chambered implant, have been tried to modify the surface topography or the chemistry of the implants. Reports have shown a limited effect on the osseointegration level along its outer surface, perhaps, because of the physically restricted diffusion [39].

Sul et al [2] presented a comparative study of experimental implants with attached Mg ions and found a stronger bone response despite an sa and an sdr of 0.78µm and 27% respectively. They assumed it to be due to the chemical bonding of the Mg implants. However, the presence of the chemical bonding remains difficult to prove.

### Polymeric Implants

Polymeric implants [4] in the form of polymethylmethacrylate and polytetrafluoroethylene were first used in the 1930s. Their use is now confined to the components of the implants.

### PEEK implants

PEEK poly etheretherketone [40] has been increasingly employed as a biomaterial for orthopaedic and spinal implants. Their

osseointegration capacity has been established beyond doubt, but it is not clear whether these implants are suitable for a specific situation with low functional and high aesthetic demands or as completely viable alternatives to titanium in all situations. They were first used for dental applications (1992) as aesthetic abutments and later as implants. They are relatively a new family of high temperature, thermoplastic polymers which consist of an aromatic backbone molecular chain which is interconnected by ketone and ether functional groups. The chemical structure of the aromatic ketones confers stability at higher temperatures, resistance to chemical and radiation damage, compatibility with many reinforcing agents as glass and carbon fibres and a greater strength on a per mass basis than many metals. These implants are available in three fundamentally different designs; TAU, THETA and IOTA for different bone volumes and densities. They have satisfactory bioinertness and fracture resistance and have been shown to promote human osteoblast cell growth and proliferation, making it osteoconductive. This study has also reported a successful immediate modification of a single piece implant with abutment, as polymeric composites do not generate heat.

## SUMMARY

Some studies [41] support the hypothesis that in case of a favourable bone quality implant, the surface roughness plays a minor role. A positive influence of the moderate rough surfaces on the early loading concepts has been suggested by many groups. A positive effect of the surface roughness has been observed in poor quality bone, but the pivotal proof of this effect is still lacking, according to some studies. Some indications which support the selection of HA coated implants over metallic implants include, the need for a greater bone implant interface contact and the ability to be placed in type IV bone, fresh extraction sites and newly grafted sites [4].

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**FINANCIAL OR OTHER COMPETING INTERESTS:**

None.

Date Of Submission: **Nov 17, 2011**  
 Date Of Peer Review: **Jan 23, 2012**  
 Date Of Acceptance: **Jan 24, 2012**  
 Date Of Publishing: **Apr 15, 2012**