

Research article

Identification card and codification of the chemical and morphological characteristics of 62 dental implant surfaces. Part 4: Resorbable Blasting Media (RBM), Dual Acid-Etched (DAE), Subtractive Impregnated Micro/Nanotextured (SIMN) and related surfaces (Group 2B, other subtractive process)

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Abstract

Background and objectives. Dental implants are commonly used in dental therapeutics, but dental practitioners only have limited information about the characteristics of the implant materials they take the responsibility to place in their patients. The objective of this work is to describe the chemical and morphological characteristics of 62 implant surfaces available on the market and establish their respective Identification (ID) Card, following the Implant Surface Identification Standard (ISIS). In this fourth part, surfaces produced through other subtractive processes (resorbable blasting media RBM, dual acid-etching DAE, subtractive impregnation micro/nanotexturization SIMN and others) were investigated.

Materials and Methods. Twenty different implant surfaces were characterized: MTX (Zimmer, Carlsbad, CA, USA), Biohorizons RBT (Biohorizons, Birmingham, AL, USA), OsseoFix (ADIN, Afula, Israel), Ossean (Intra-Lock, Boca Raton, Florida, USA), Blossom Ossean (Intra-Lock, Boca Raton, Florida, USA), Osstem RBM (Osstem implant Co., Busan, Korea), Ossean G23 ELI (Intra-Lock, Boca Raton, Florida, USA), SBM body (Implant Direct LLC, Calabasas, CA, USA), MegaGen RBM (MegaGen Co., Seoul, Korea), DIO BioTite-M (DIO Corporation, Busan, Korea), Blue Sky Bio RBM (Blue Sky Bio, Grayslake, IL, USA), Anthogyr BCP (Anthogyr, Sallanches, France), Shinhung RBM+ (Shinhung Co., Seoul,

Korea), Neobiotech CMI (Neobiotech Co., Seoul, Korea), Osseospeed (AstraTech, Mölndal, Sweden), 3I OsseoTite (Biomet 3I, Palm Beach Gardens, FL, USA), 3I OsseoTite 2 (Biomet 3I, Palm Beach Gardens, FL, USA), Neoss ProActive (Neoss Ltd, Harrogate, UK), BTI Interna (Biotechnology Institute, Vitoria, Spain), Winsix WMRS (BioSAF IN, Ancona, Italy). Three samples of each implant were analyzed. Superficial chemical composition was analyzed using XPS/ESCA (X-Ray Photoelectron Spectroscopy/Electron Spectroscopy for Chemical Analysis) and the 100nm in-depth profile was established using Auger Electron Spectroscopy (AES). The microtopography was quantified using optical profilometry (OP). The general morphology and the nanotopography were evaluated using a Field Emission-Scanning Electron Microscope (FE-SEM). Finally, the characterization code of each surface was established using the ISIS, and the main characteristics of each surface were summarized in a reader-friendly ID card.

Results. From a chemical standpoint, in the 20 different surfaces of this group, 12 were based on a commercially pure titanium (grade 4) and 8 on a titanium-aluminium alloy (grade 5 or grade 23 ELI titanium). 16 surfaces presented different forms of chemical impregnation (most frequently with calcium phosphate CaP) and one surface presented a CaP particles discontinuous coating of the titanium core. 15 surfaces presented different degrees of inorganic pollutions, and 4 presented a significant organic pollution overcoat. Only 5 surfaces presented no pollution (Osseospeed, Ossean, Blossom Osseans and Blue Sky Bio). From a morphological standpoint, all surfaces were microrough, with different microtopographical aspects and values. 16 surfaces were smooth on the nanoscale, and therefore presented no significant and repetitive nanostructures. Four implants only were nanorough (Osseospeed, Ossean, Blossom Osseans), following a SIMN production process. One surface (ProActive) was covered with extended cracks all over the surface. 17 surfaces were homogeneous and 3 heterogeneous. Only 3 surfaces were fractal.

Discussion and Conclusion. The ISIS systematic approach allowed to gather the main characteristics of these commercially available products in a clear and accurate ID card. The RBM surfaces have specific morphological characteristics (microrough, CaP impregnation) and are frequently used in the industry, and many other technologies exist. All these surfaces presented different designs, and pollutions were often detected. Users should be aware of these specificities if they decide to use these products. Finally, the SIMN surfaces appeared as an interesting evolution for the various subtractive technologies, to develop specific chemical modification, microtexture and nanotexture.

Keywords. Dental implant, nanostructure, osseointegration, surface properties, titanium.

1. Introduction

Dental implants are commonly used in daily dental therapeutics. Each implant system can be defined by several key characteristics that determine its biological behavior, particularly the chemical and morphological characteristics of each implant surface [1]. Implant users have however very limited information about these characteristics when they choose the implant system they take the responsibility to use in their patients [1]. The surface characteristics are often advertised by the dental implant companies in order to promote their products [2], but most data remain very commercial and without certified evaluation and disclosure of the surfaces characteristics [3]. In 2010, a first standard of characterization, terminology, classification and codification of dental implant surfaces was published [1]. This standard is based on the use of standardized tools of analysis to establish a detailed characterization and identification card for each osseointegrated implant surface

[4,5]. This card describes the surface chemical composition and morphological characteristics of each surface. This standardized codification system allows to clarify the identity of each surface and to easily sort their differences [6]. In this series of 5 articles, we proposed an update and a final form of the standard proposed in 2010 [1], based on the feedback of recent experience, and 62 implant surfaces were characterized following this protocol. This final system, termed ISIS (Implant Surface Identification Standard) may be used as an official international standard in the future.

The second category of methods (arbitrarily termed Group 2) to create a dental implant surface is to carve the morphology of the surface on the core material using a subtractive process, that can be associated with some chemical modifications. All the surfaces of this category are presenting various levels of microroughness [4]. The most common version of this approach is the combination of sand-blasting and acid-etching (SLA type). However several other subtractive processes are frequent in the industry [7], particularly the carving of the surface through the use of Resorbable Blasting Media (RBM)[8], the use of Dual Acid-Etching (DAE)[9] or other combinations of blasting and/or etching processes, particularly for Subtractive Impregnation Micro/Nanotexturization (SIMN)[10], which are all regrouped in the Group 2B.

The use of RBM is one of the most classical subtractive techniques used for dental implant surfaces [8]. The titanium core material is blasted with microparticles of hydroxyapatite or similar calcium phosphate (CaP) particles (i.e. the resorbable blasting media) to carve a microtopography on the surface, and then washed softly (in general) to remove only the blasting residues. The blasting media is expected to leave a significant quantity of CaP impregnated in the titanium external layer, to improve the osseointegration. This method is often associated with the company Zimmer [8], as it was the largest company promoting this surface, but nowadays many companies are using this technology or equivalent [4].

Dual Acid-Etching is also a quite classical technique, as it was promoted by some major companies (Biomet 3I)[9] during many years. In this process, the surface is carved only through the use of several etching preparations, what creates a typical relatively small microroughness. Many combinations of acid-etching are possible, and “Combined Acid-Etching” (CAE) would be a better acronym than DAE. This approach is however not widely spread in the industry, as most companies promoted higher microroughness.

Finally, other combinations of blasting and/or etching exist, particularly for the development of Subtractive Impregnation Micro/Nanotexturization (SIMN). A well known example of SIMN is the surface carving through blasting with TiO_2 particles and etching with hydrofluoric acid (Astra Osseospeed)[11]. Some combinations using RBM technology also allow to perform a Subtractive Impregnation Micro/Nanotexturization (Intra-Lock Ossean)[10]. These 2 examples are famous and the technologies still relatively secret: each surface design in this category is - at this time - unique and specific to its inventor company only.

In this fourth part, the chemical and morphological characteristics of 20 implant surfaces (available on the market) from the group 2B were investigated and described through a simple and clear identification (ID) card for each surface, following the ISIS system terminology and classification. The group 2B gathered surfaces produced through a subtractive processing to carve the surface morphology on the core material, using RBM, DAE, SIMN and other related techniques (except SLA type surfaces).

2. Materials and Methods

2.1. Samples

Twenty different implant surfaces of the Group 2B have been investigated: MTX (Zimmer, Carlsbad, CA, USA), Biohorizons RBT (Biohorizons, Birmingham, AL, USA), OsseoFix (ADIN, Afula, Israel), Ossean (Intra-Lock, Boca Raton, Florida, USA), Blossom Ossean (Intra-Lock, Boca Raton, Florida, USA), Osstem RBM (Osstem implant Co., Busan, Korea), Ossean G23 ELI (Intra-Lock, Boca Raton, Florida, USA), SBM body (Implant Direct LLC, Calabasas, CA, USA), MegaGen RBM (MegaGen Co., Seoul, Korea), DIO BioTite-M (DIO Corporation, Busan, Korea), Blue Sky Bio RBM (Blue Sky Bio, Grayslake, IL, USA), Anthogyr BCP (Anthogyr, Sallanches, France), Shinhung RBM+ (Shinhung Co., Seoul, Korea), Neobiotech CMI (Neobiotech Co., Seoul, Korea), Osseospeed (AstraTech, Mölndal, Sweden), 3I OsseoTite (Biomet 3I, Palm Beach Gardens, FL, USA), 3I OsseoTite 2 (Biomet 3I, Palm Beach Gardens, FL, USA), Neoss ProActive (Neoss Ltd, Harrogate, UK), BTI Interna (Biotechnology Institute, Vitoria, Spain), Winsix WMRS (BioSAF IN, Ancona, Italy). Three samples were used per implant system, and their reference and batch were reported in their respective ID card. All samples were obtained on the market by the various partners of this study (private clinicians or academics), without communication on the purpose of this study or interferences from the companies, except the MegaGen and Blue Sky Bio implants that were offered by the companies.

2.2. Chemical analyses

The chemical characteristics of the surfaces have been evaluated using 2 techniques of investigation.

The superficial atomic composition and chemistry of all the samples have been evaluated accurately through X-Ray Photoelectron Spectroscopy (XPS)/Electron Spectroscopy for Chemical Analysis (ESCA) using a PHI Quantum 2000 instrument (Physical Electronics Inc., Chanhassen, MN, USA; analytical parameters: monochromatic X-ray source AlK α 1486.6eV, acceptance angle $\pm 23^\circ$, take-off angle 45° , charge correction C1s 284.8 eV), on a 100 μ m diameter analysis area located between the second and third threads of each sample. This technique allowed to analyze surface chemistry of a 5-10nm thick superficial layer. Detailed chemical composition was reported in percentages in each ID card.

The in-depth analysis of the chemical composition of the external surface layer was performed through Auger Electron Spectroscopy (AES) using a PHI 670 Scanning Auger Nanoprobe instrument (Physical Electronics Inc., Chanhassen, MN, USA; Electron Beam Energy 10keV, 20nA; Tilt 30° to sample normal) on a very small analysis area (30nm in diameter) located in the middle of the cutting edge flat area (or an equivalent flat part, depending on the implant macrodesign) of each implant. The in-depth chemical profile was established down to 100nm, using sputtering cycles with a 4keV Ar+ source (Ar+ etching rate for TiO $_2$: 3.3nm/min). Two in-depth profiles were established per sample. The analysis area being very small, the 2 spots were very precisely located, respectively on a peak and in a valley of the surface microtopography. One in-depth profile graph was reported in each ID card.

2.3. Morphological analysis

The morphological characteristics of the surfaces have been evaluated using 2 techniques of investigation.

The general morphology of the surfaces has been evaluated and described separately by 2 independent teams with a Field Emission-Scanning Electron Microscope (FE-SEM, Hitachi S-4700, Hitachi HTA, Pleasanton, CA, USA) up to x200 000 magnification. All the areas of the implants have been carefully examined, from the macroscale to the nanoscale. This examination allowed to highlight various morphological characteristics of the surfaces (cracks, blasting residues, homogeneity) and to determine the kind of nanotopography of each sample (nanosmooth, nanorough, nanopatterned or nanoparticled). In each ID card, a first x1000 magnification picture was provided to illustrate the general aspect of the microtopography of each surface (it replaced the interferometer three-dimensional reconstruction picture used in the early version of the ISIS system)[4]. Then a second x5000 magnification picture was added to illustrate in more details the morphological characteristics of the surfaces (micropores, cracks, blasting residues for example). Finally, a x100 000 magnification picture was added to show the nanotopography of each surface, a small picture if nanosmooth and a wider picture if some nanopatterns or nanoroughness could be observed.

The microtopography has been quantified using an optical profilometer (OP, ContourGT-X8, Bruker Corporation, Tucson, Arizona, USA). Three spots of analysis were selected on the flat cutting edge (or similar area in the lower part) of the implant and the corrected mean values (and standard deviations) calculated on these large areas were placed as reference values in each ID card. Another spot of analysis was selected in the middle of the implant between threads to serve as a control value for homogeneity check. One final set of experimental analyses was performed following the guidelines used in the previous classification study [4], i.e. evaluating the topography on the top, valley and flank of 3 successive threads and calculating the corrected mean values of these large areas, to serve as a supplementary control evaluation. The dimensions of the analyzed areas were 200x260 microns most time, but the area could be a little bit smaller depending on the implant macrogeometry. Images were post-processed with a 50x50 μ m Gaussian filter.

Eighteen topographical parameters were assessed but only 2 were considered as significant for the classification of the surface characteristics: the Sa (height deviation amplitude of the microtopography, also called « roughness average ») and the Sdr% (hybrid parameter integrating both the number and height of peaks of the microtopography, also called « developed interfacial area ratio »). The Sa is an important and frequent parameter for the comparison of surfaces and was already used in other classifications. The Sdr% is calculated as a developed area ratio relative to a flat plane baseline. For a totally flat surface, Sdr = 0%. When Sdr = 100%, it means that the roughness of a surface doubled its developed area. These Sa and Sdr% values allowed to classify the microtopography, following the system developed in the ISIS.

3. Results

3.1. General results

From a chemical standpoint, in the 20 different surfaces of this group, 12 were based on a commercially pure titanium (grade 4) and 8 on a titanium-aluminium alloy (grade 5 or grade 23 ELI titanium). 16 surfaces presented different forms of chemical impregnation (most frequently with calcium phosphate CaP) and one surface presented a CaP particles discontinuous coating of the titanium core. 15 surfaces presented different degrees of

inorganic pollutions, and 4 presented a significant organic pollution overcoat. Only 5 surfaces presented no pollution (Osseospeed, Ossean, Blossom Osseans and Blue Sky Bio).

From a morphological standpoint, all surfaces were microrough, with different microtopographical aspects and values. 16 surfaces were smooth on the nanoscale, and therefore presented no significant and repetitive nanostructures. Four implants only were nanorough (Osseospeed, Ossean, Blossom Osseans), following a SIMN production process. One surface (ProActive) was covered with extended cracks all over the surface. 17 surfaces were homogeneous and 3 heterogeneous. Only 3 surfaces were fractal.

Finally, data were gathered and synthesized to build for each implant surface a detailed Identification ID card, following the ISIS methodology and format.

3.2. RBM surfaces

The 14 first surfaces of this group were blasted with Resorbable Blasting Media (RBM, in general various forms of hydroxyapatite blasting particles) and were all chemically impregnated with calcium phosphate CaP (except one covered with CaP particles). Inorganic pollutions were often detected. All surfaces had in common to be microrough (with different degrees of roughness) and 11 were nanosmooth. Only 3 were nanorough and fractal, and produced following a Subtractive Impregnation Micro/Nanotexturization (SIMN) unknown process.

Zimmer MTX (Zimmer, Carlsbad, CA, USA; **Figure 1**) was produced through blasting with a Resorbable Blasting Media (RBM, hydroxyapatite) followed by washing of the particles (RBM-blasted/washed surface), on a grade 5 titanium core. Therefore the surface was impregnated with low levels of calcium phosphate (CaP), not visible with FE-SEM but homogeneous all over the surface. Some inorganic pollution with silicon was also detected. The surface was minimally microrough, nanosmooth, and homogeneous all over the implant.

Biohorizons RBT (Resorbable Blast Texture; Biohorizons, Birmingham, AL, USA; **Figure 2**) was a RBM-blasted/washed surface on a grade 5 titanium core. Therefore the surface was impregnated with low levels of calcium phosphate (CaP), not visible with FE-SEM but homogeneous all over the surface. Some inorganic pollution with silicon was also detected. The surface was moderately microrough, nanosmooth, and homogeneous all over the implant.

OsseoFix (ADIN, Afula, Israel; **Figure 3**) was a RBM-blasted without washing surface, on a grade 5 titanium core. Because of the absence of washing, the surface was covered with a discontinuous coating of calcium phosphate particles. The surface appeared also covered with a thick organic pollution (thick carbon overcoat all over the implant). Several inorganic pollutions with silicon, fluorine and magnesium were also detected. The surface was moderately microrough, nanosmooth, and heterogeneous all over the implant.

Ossean (Intra-Lock, Boca Raton, Florida, USA; **Figure 4**) was a RBM-blasted/washed surface following a Subtractive Impregnation Micro/Nanotexturization (SIMN) unknown process. The surface was impregnated with low levels of calcium phosphate (CaP), not visible with FE-SEM but homogeneous all over the surface. No pollution was detected. The microroughness was minimal, but close to the moderate level, and was covered with a nanoroughness all over the implant. The surface was homogeneous in chemistry and topography, and could be considered as fractal following our definition.

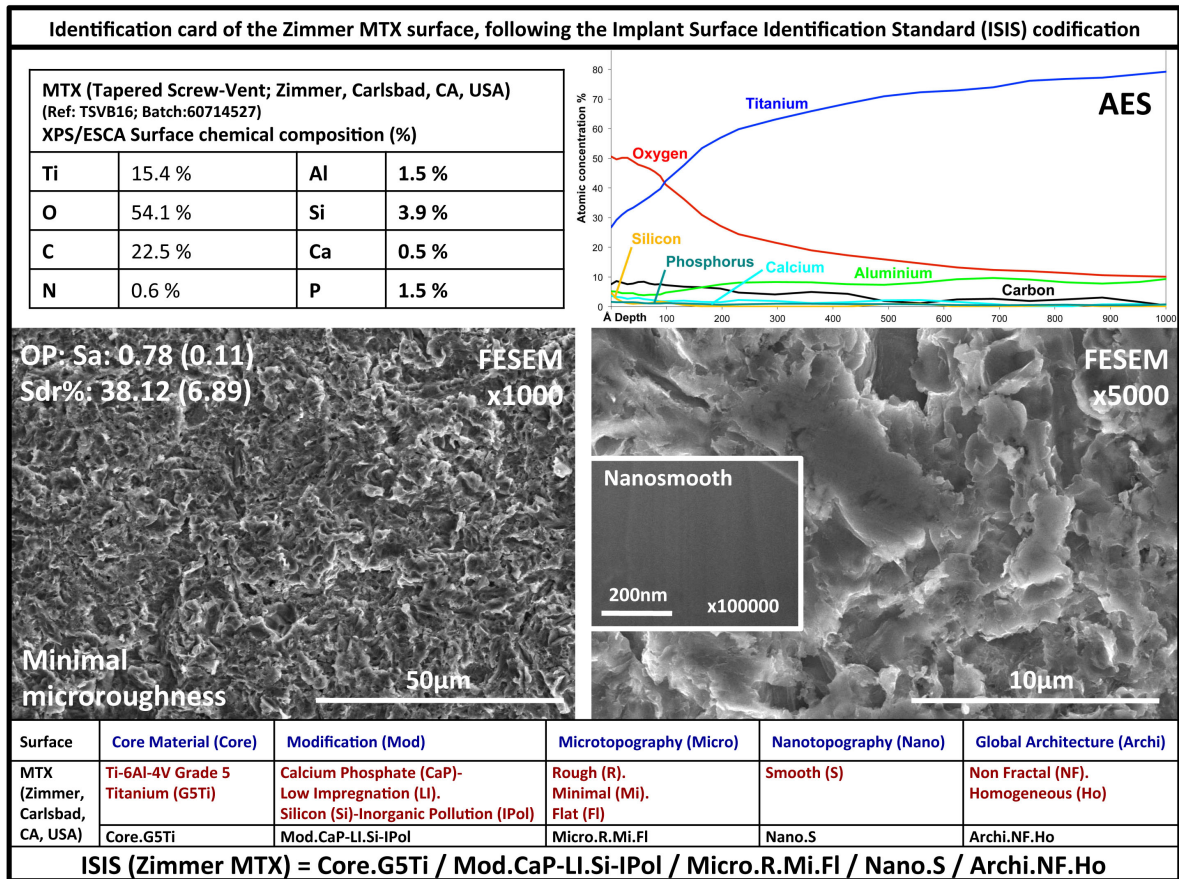


Figure 1. Identification Card of the Zimmer MTX surface.

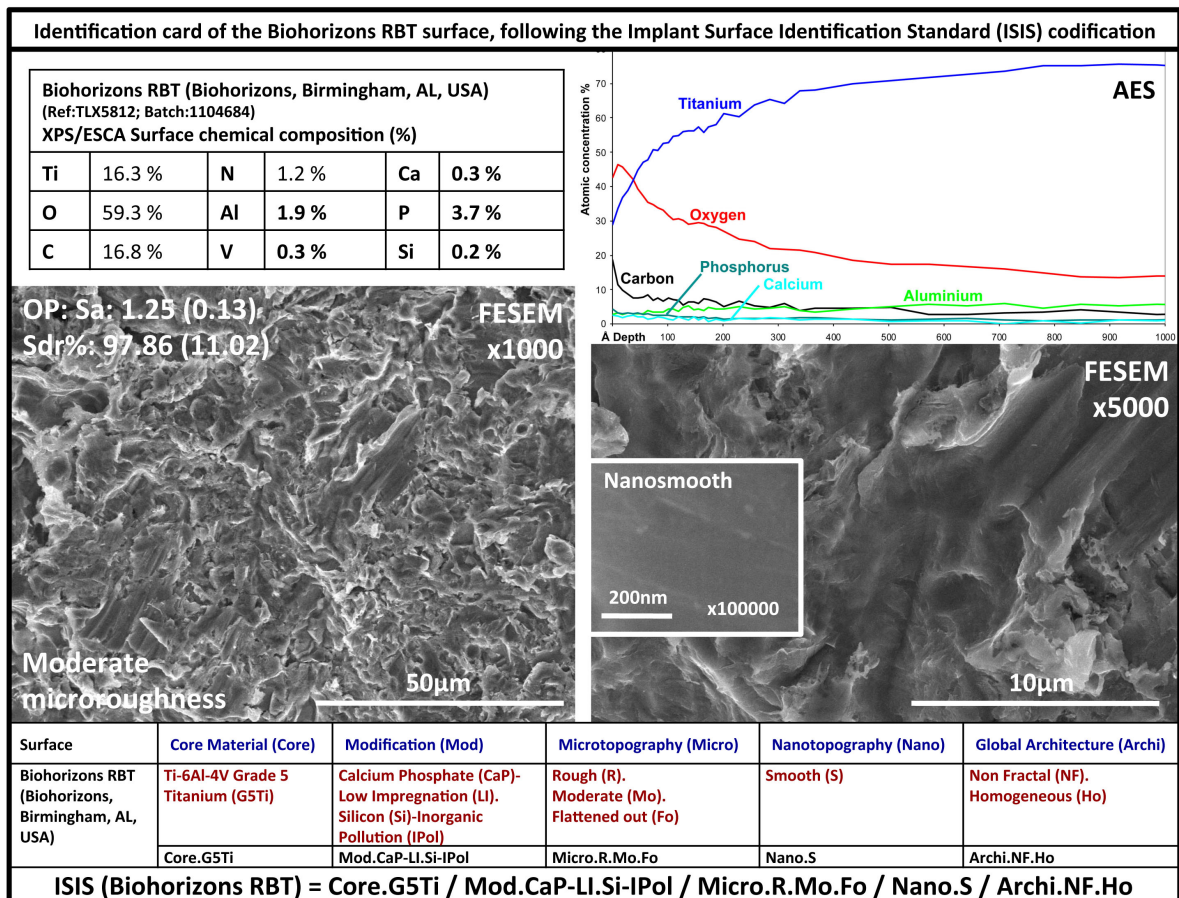


Figure 2. Identification Card of the Biohorizons RBT surface.

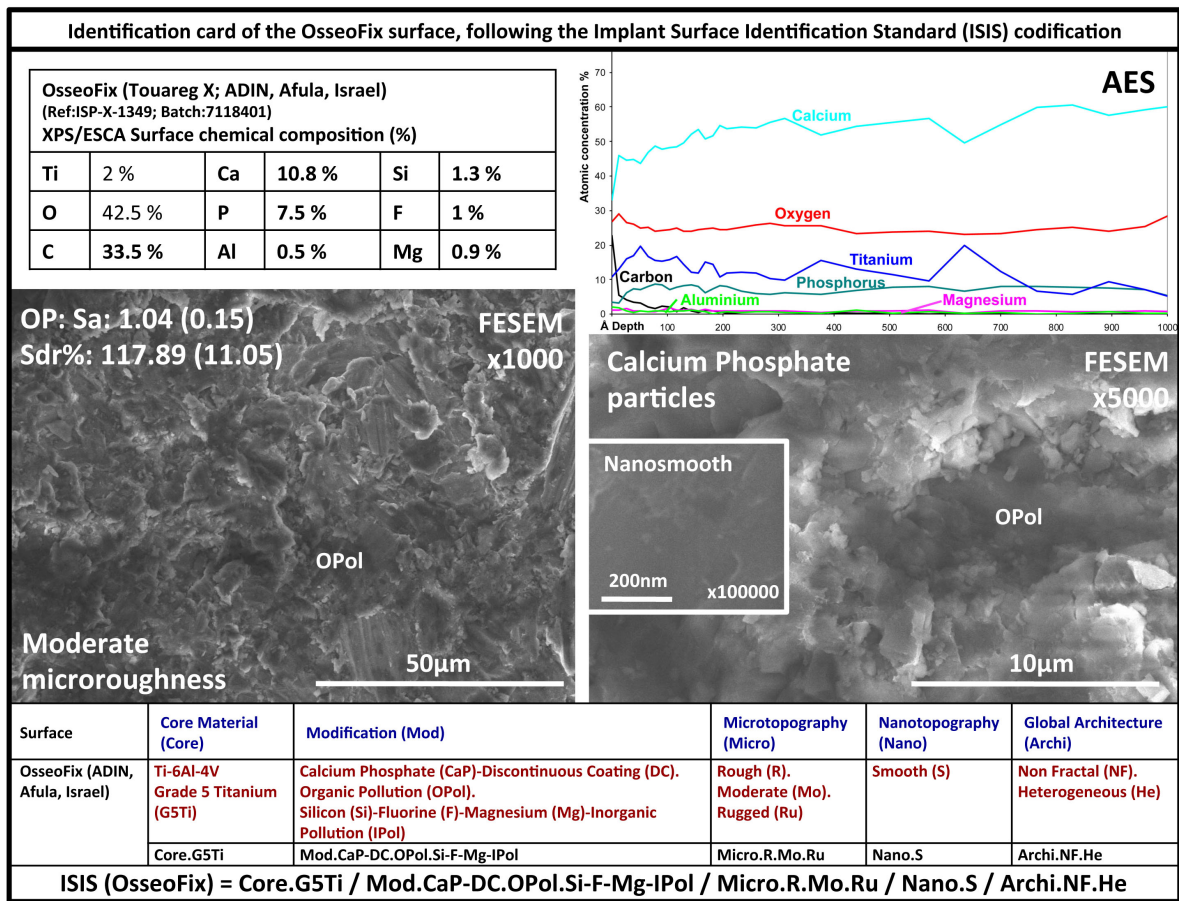


Figure 3. Identification Card of the OsseoFix surface.

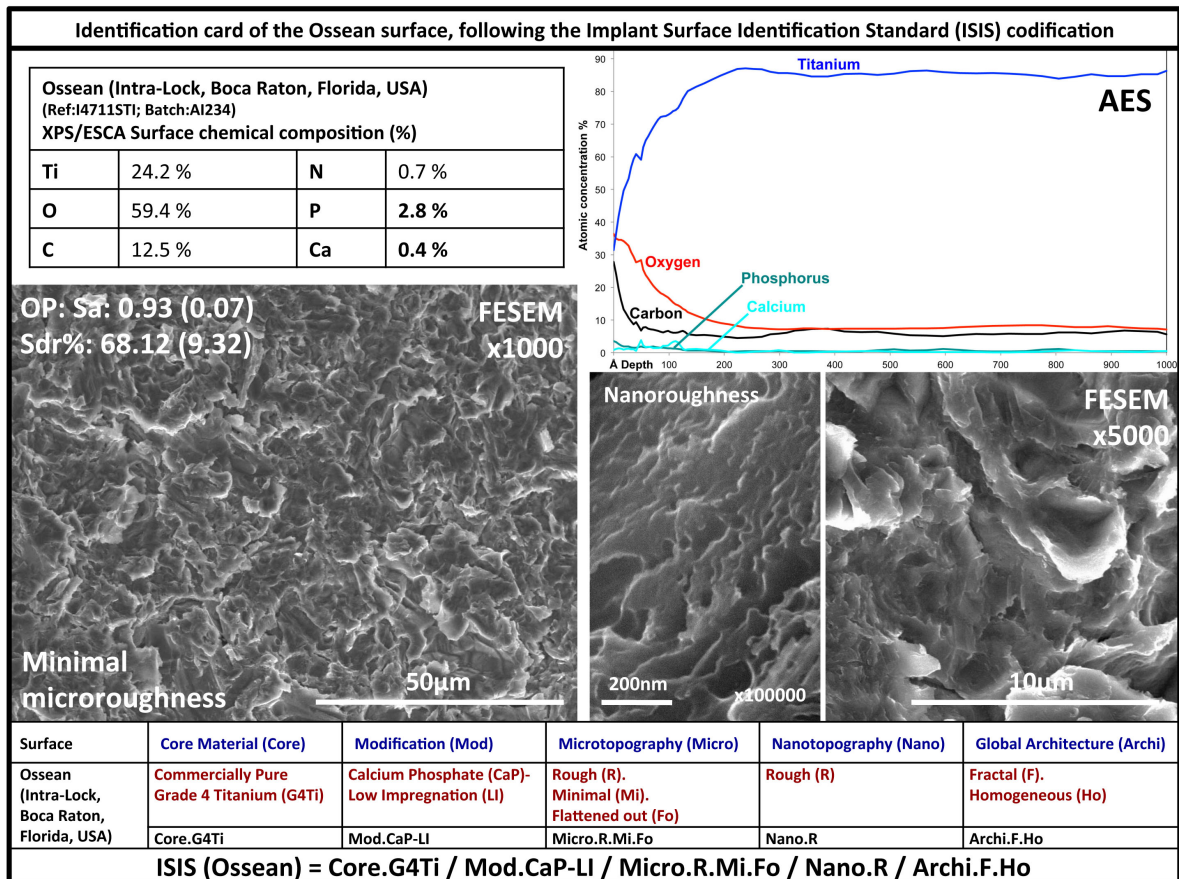


Figure 4. Identification Card of the Ossean surface.

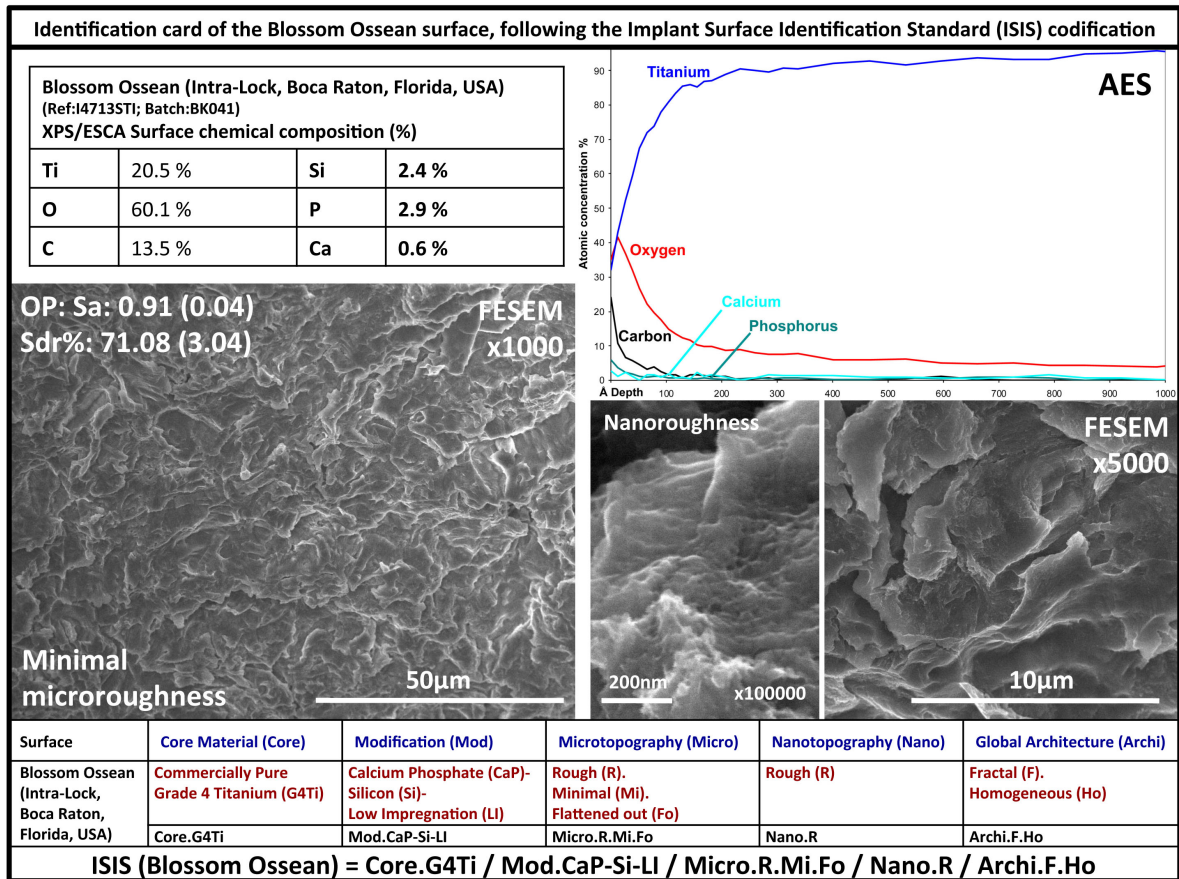


Figure 5. Identification Card of the Blossom Ossean surface.

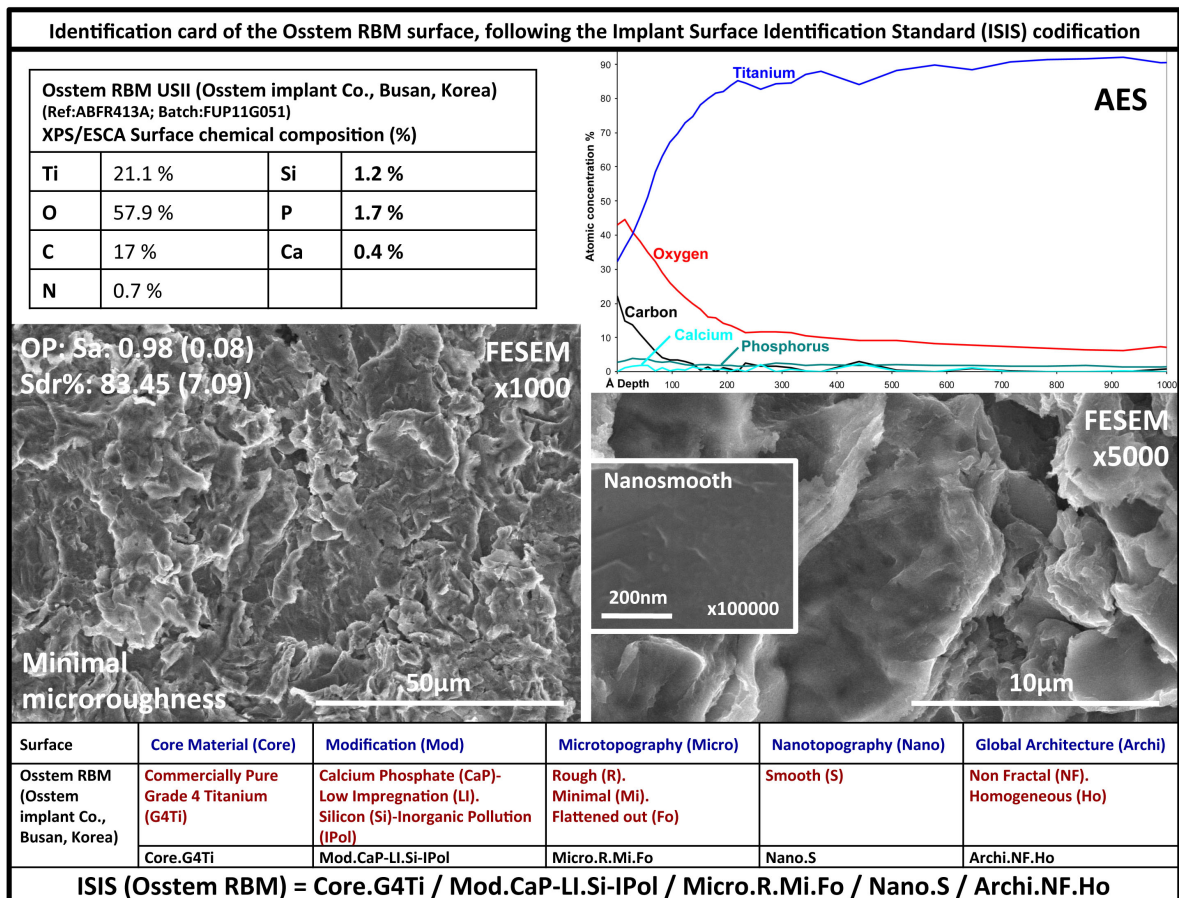


Figure 6. Identification Card of the Osstem RBM surface.

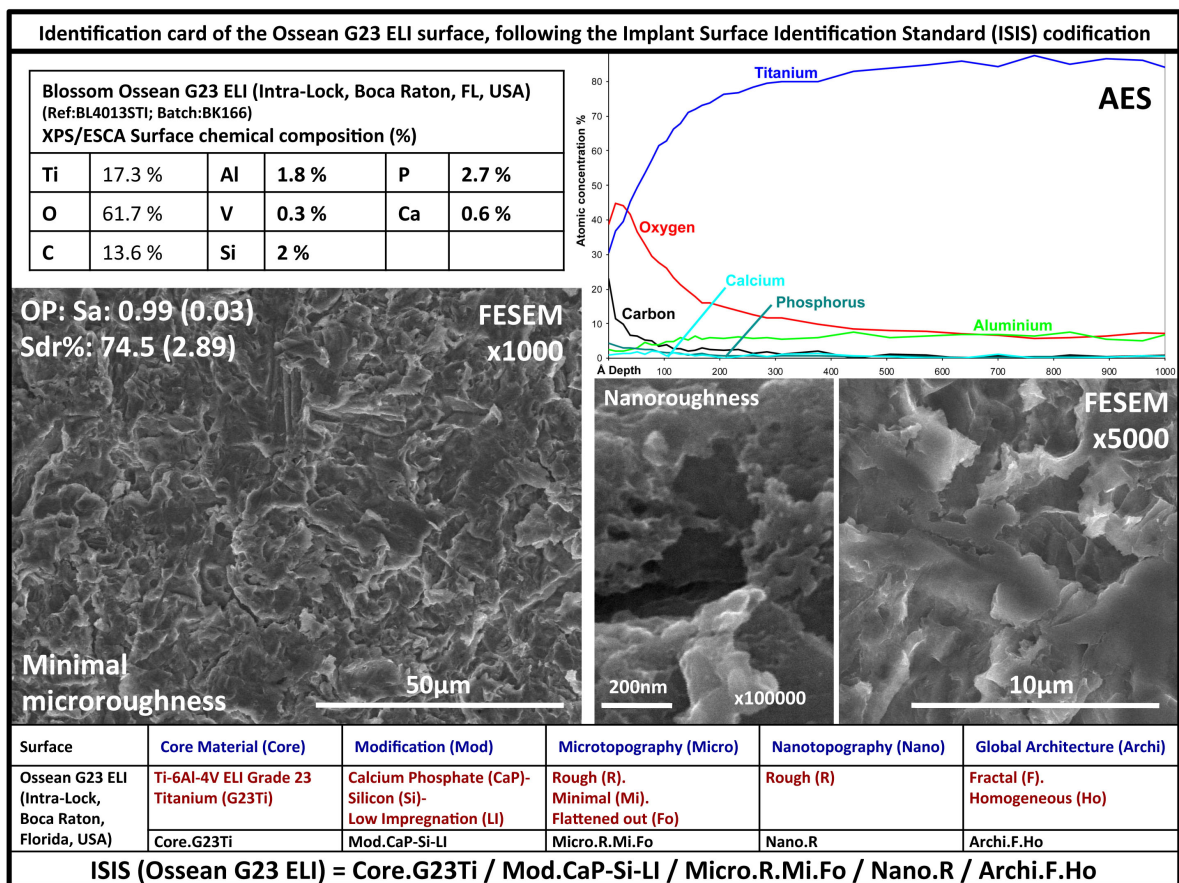


Figure 7. Identification Card of the Ossean G23 ELI surface.

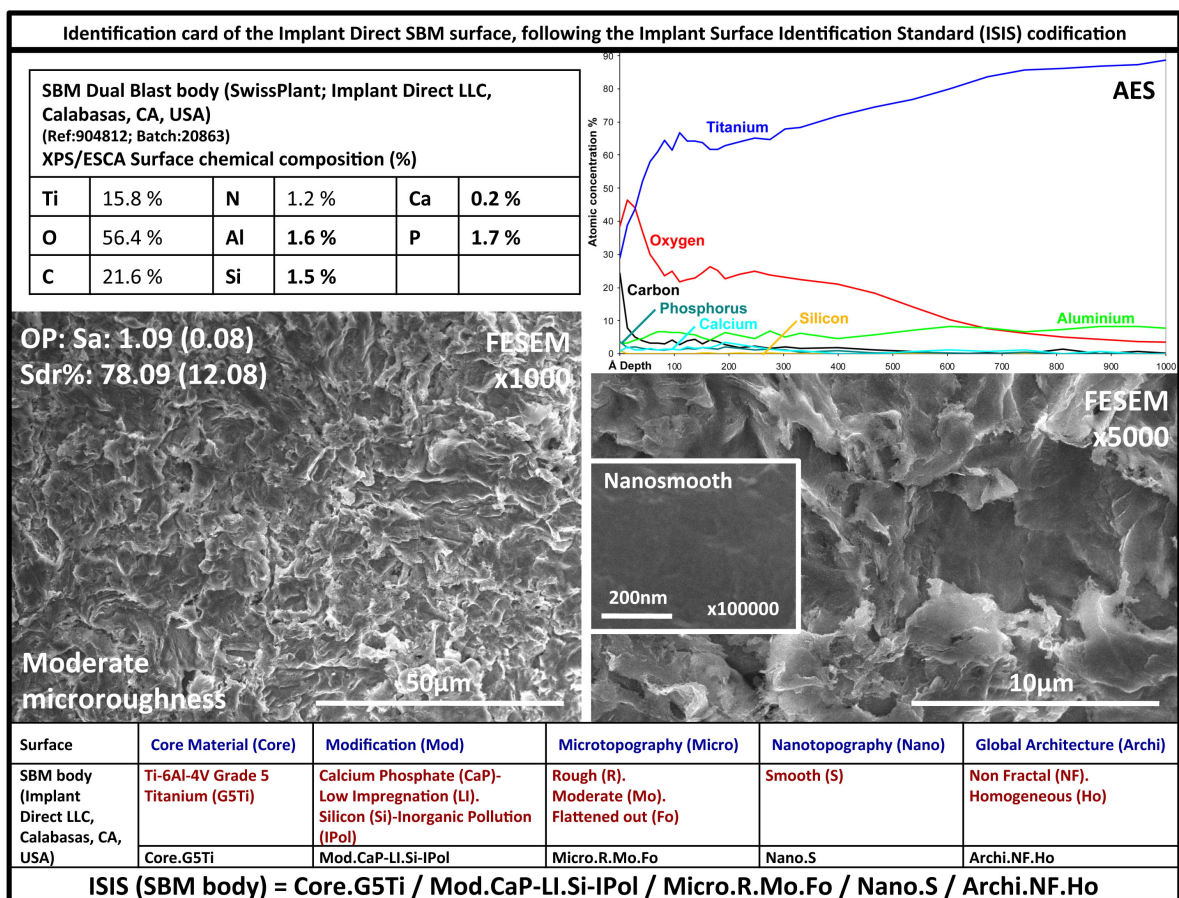


Figure 8. Identification Card of the Implant Direct SBM body surface.

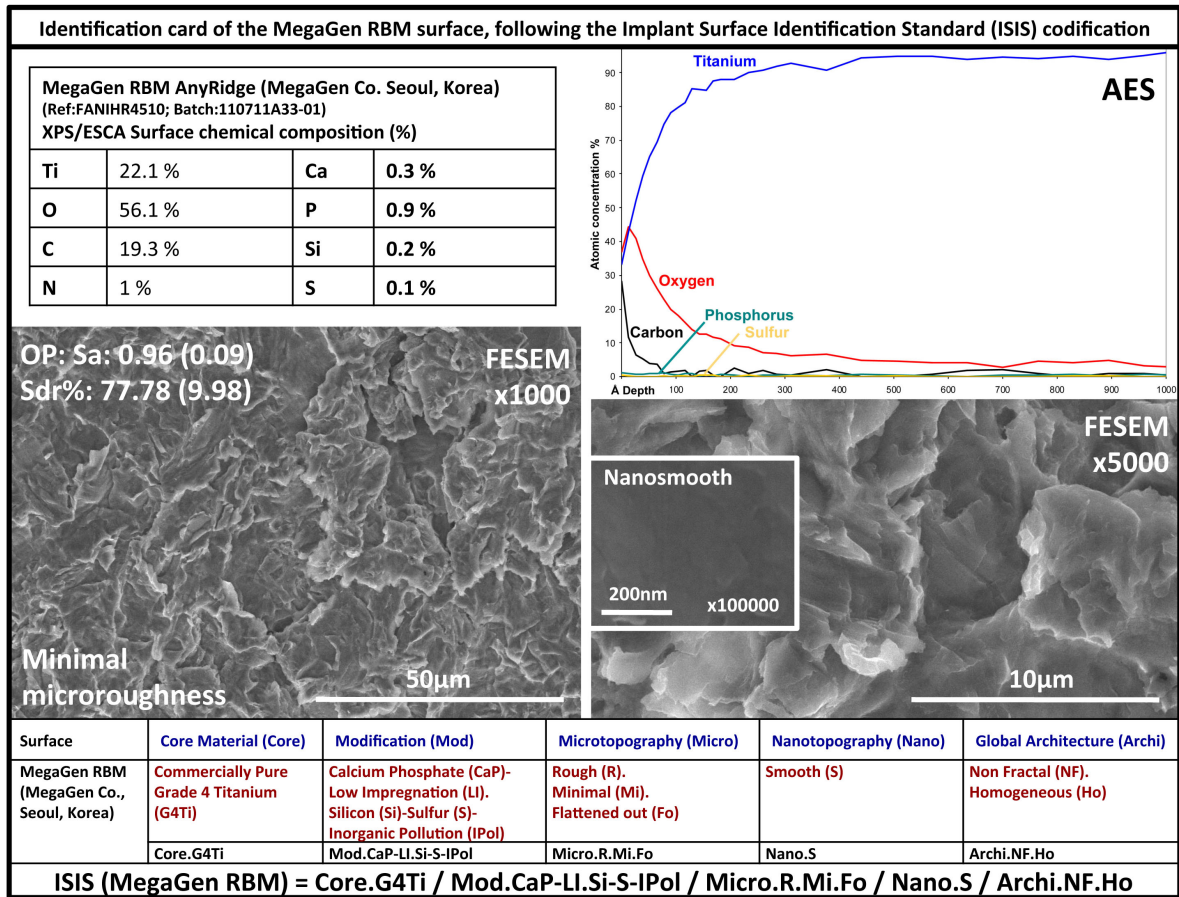


Figure 9. Identification Card of the MegaGen RBM surface.

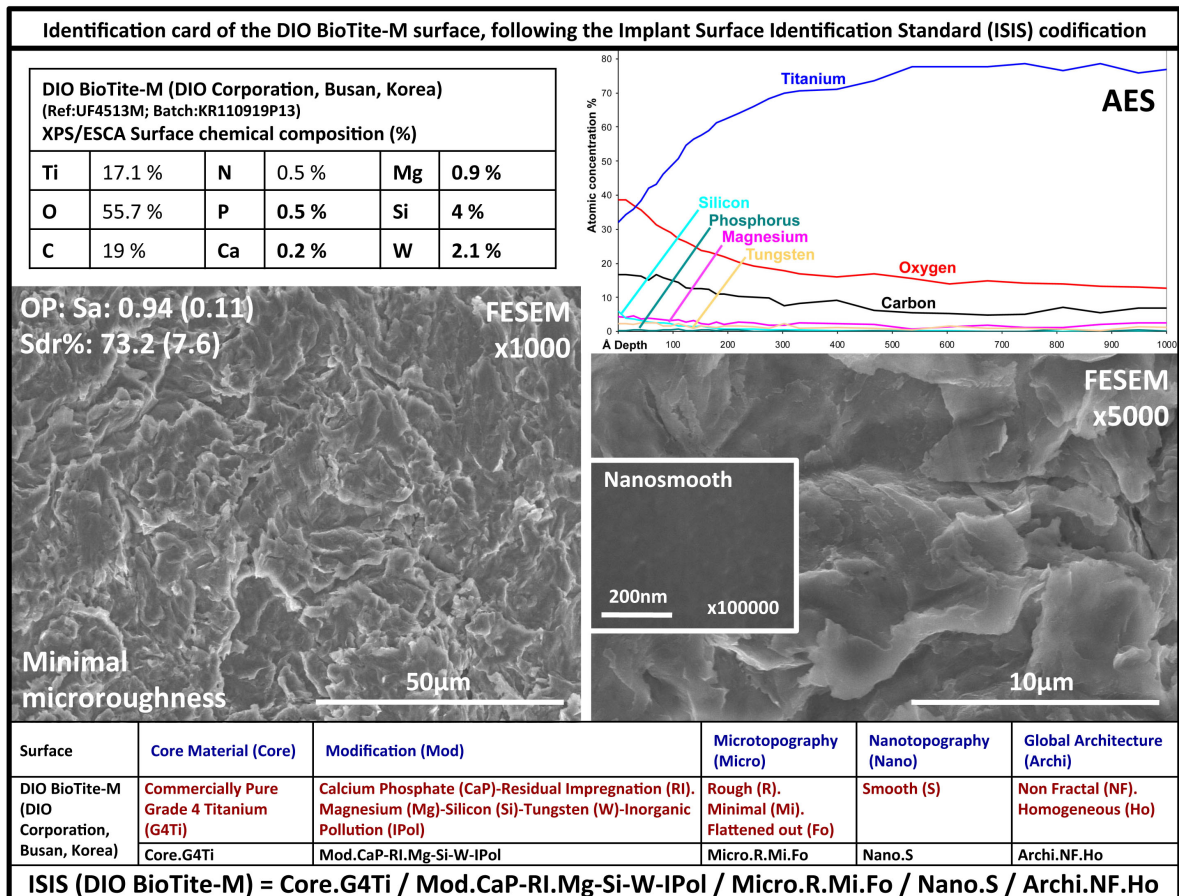


Figure 10. Identification Card of the DIO BioTite-M surface.

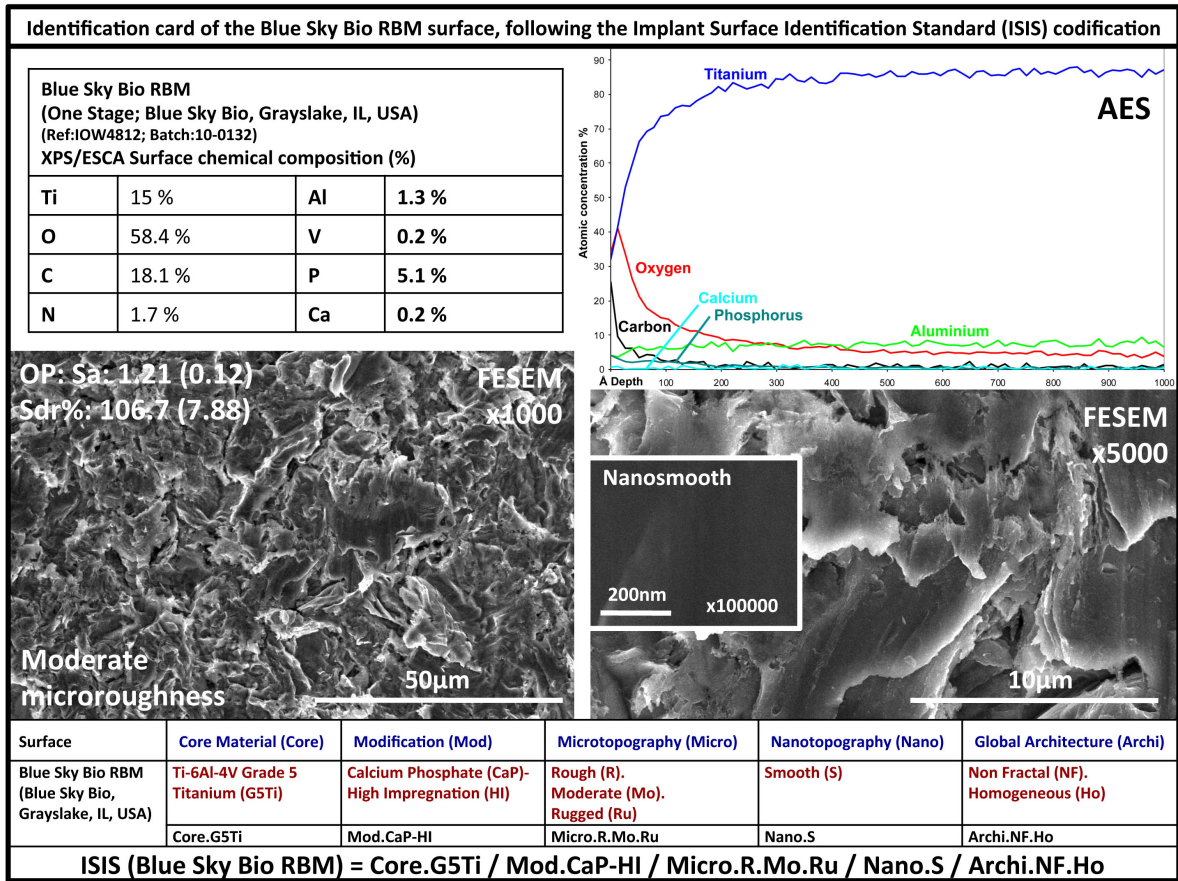


Figure 11. Identification Card of the Blue Sky Bio RBM surface.

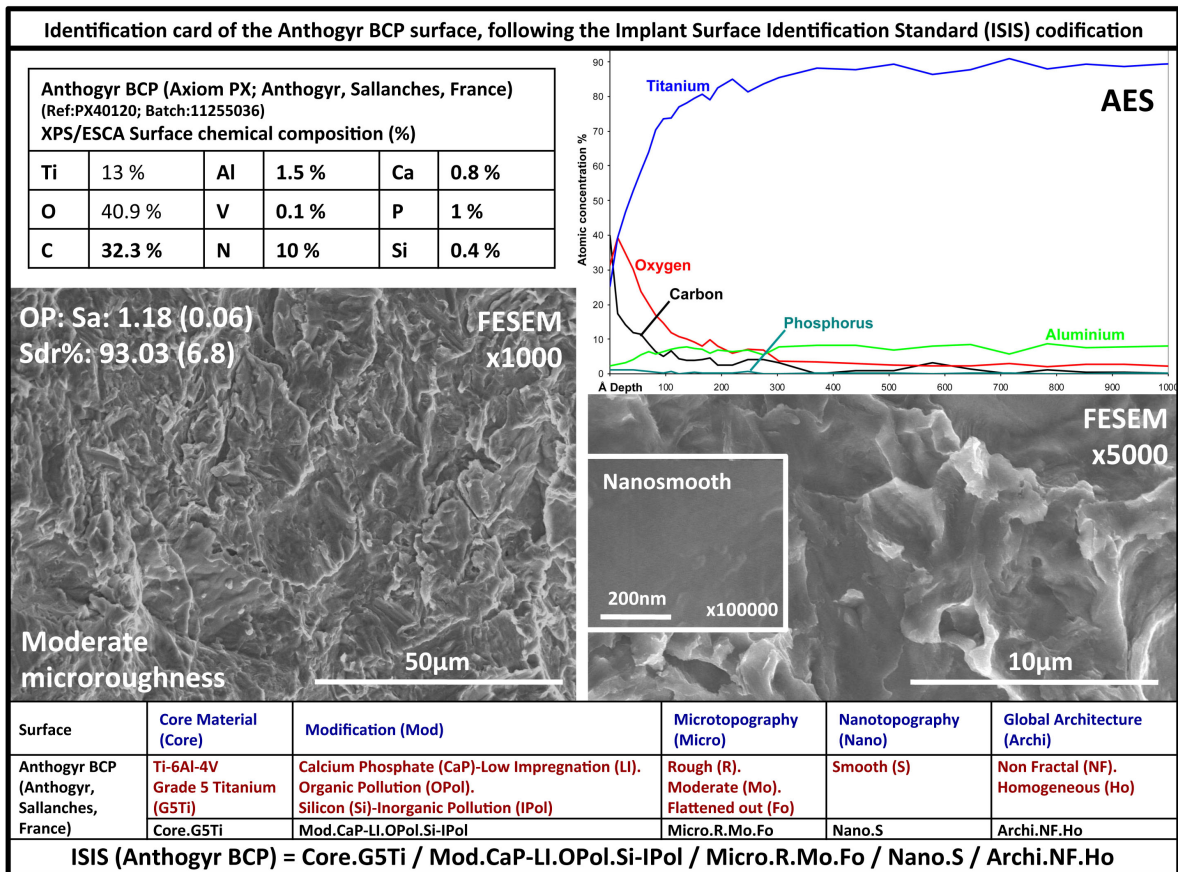


Figure 12. Identification Card of the Anthogyr BCP surface.

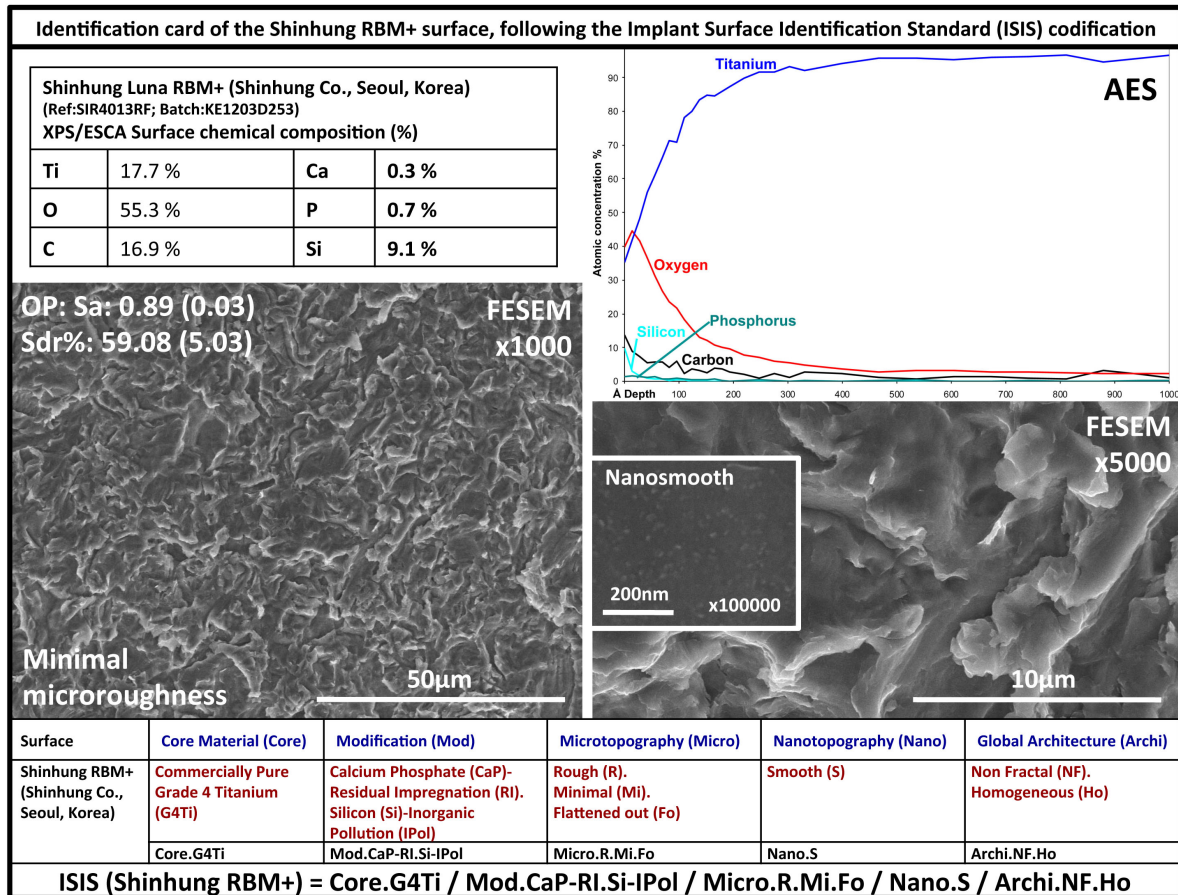


Figure 13. Identification Card of the Shinhung RBM+ surface.

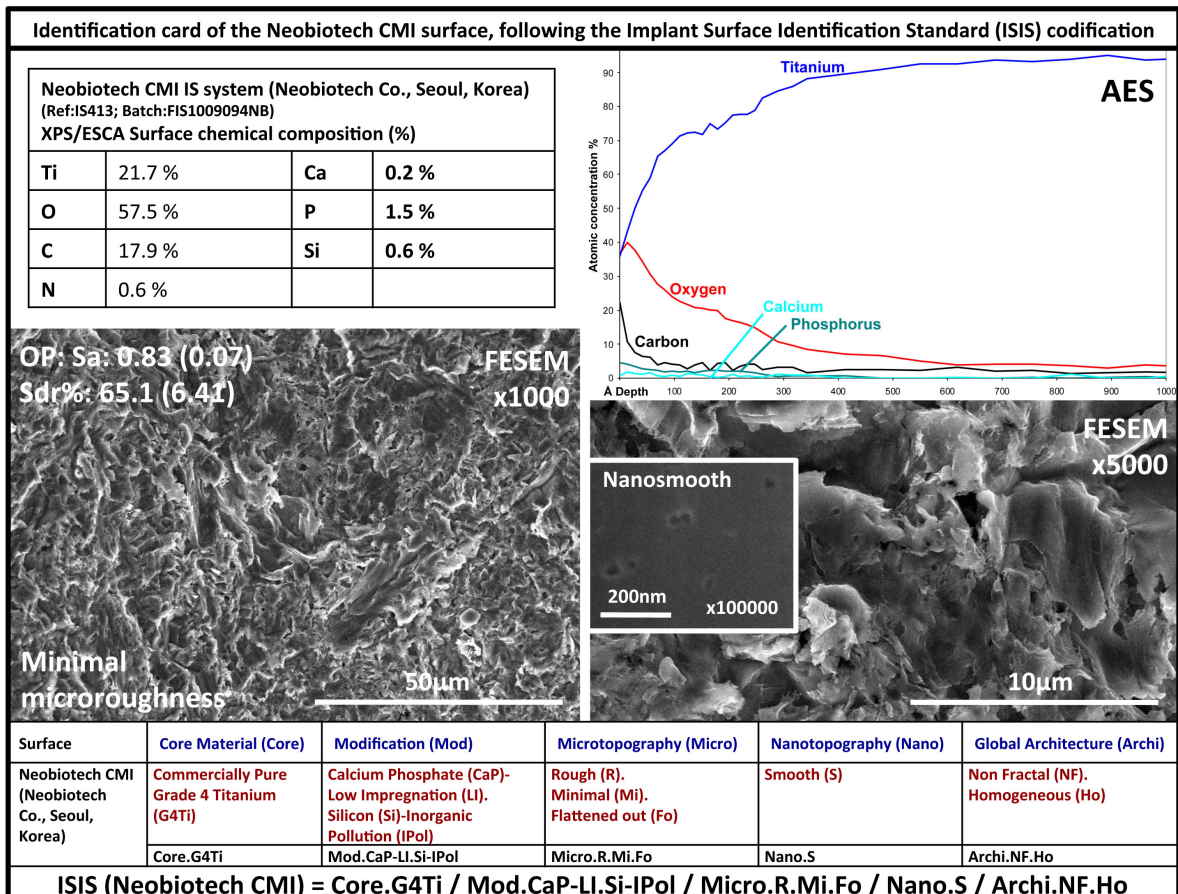


Figure 14. Identification Card of the Neobiotech CMI surface.

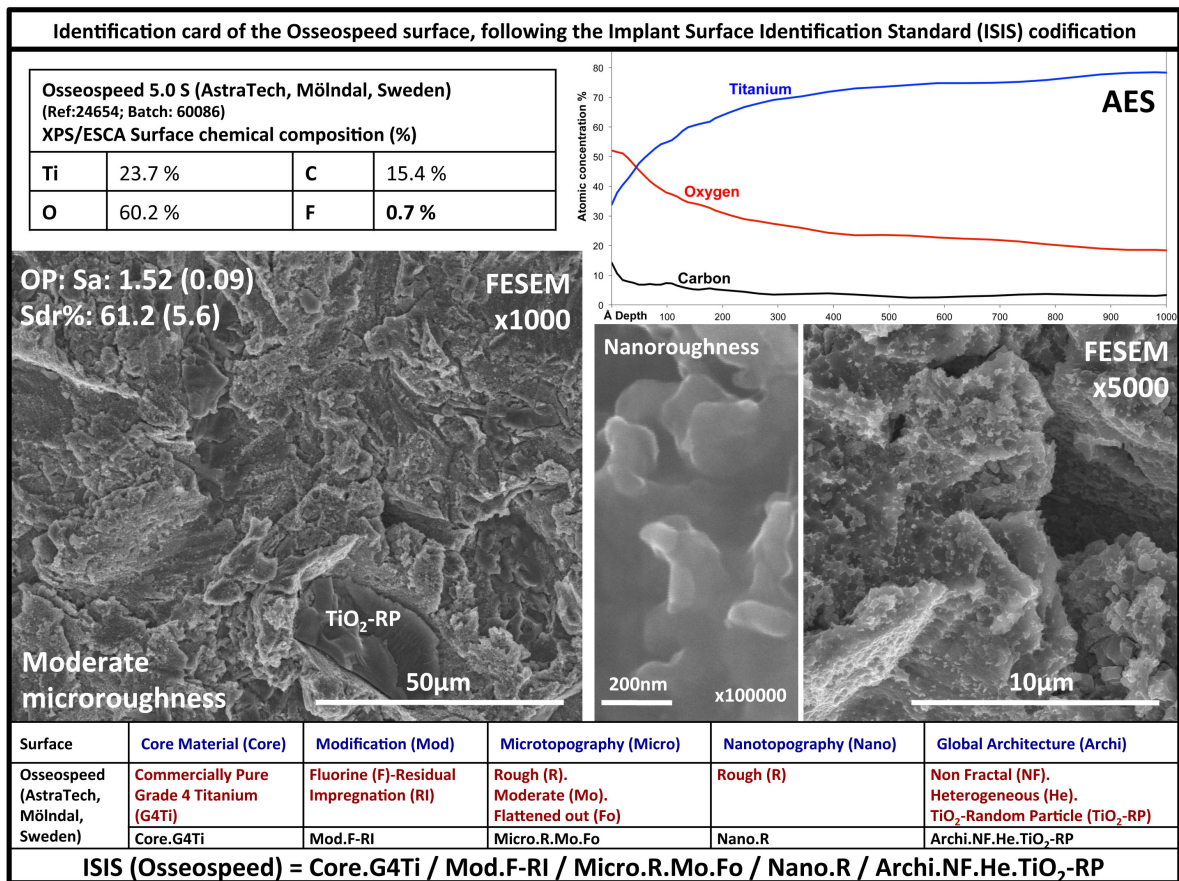


Figure 15. Identification Card of the Osseospeed surface.

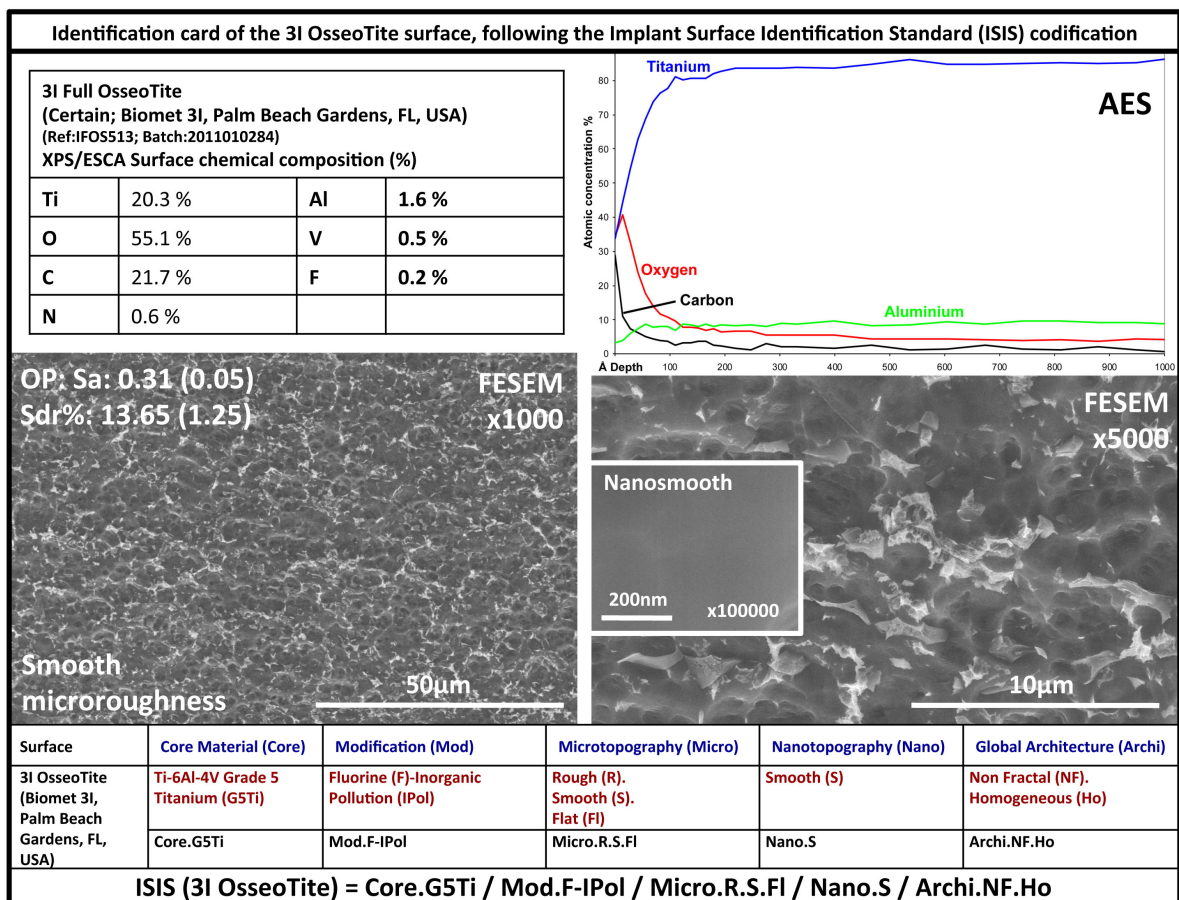


Figure 16. Identification Card of the 3I OsseoTite surface.

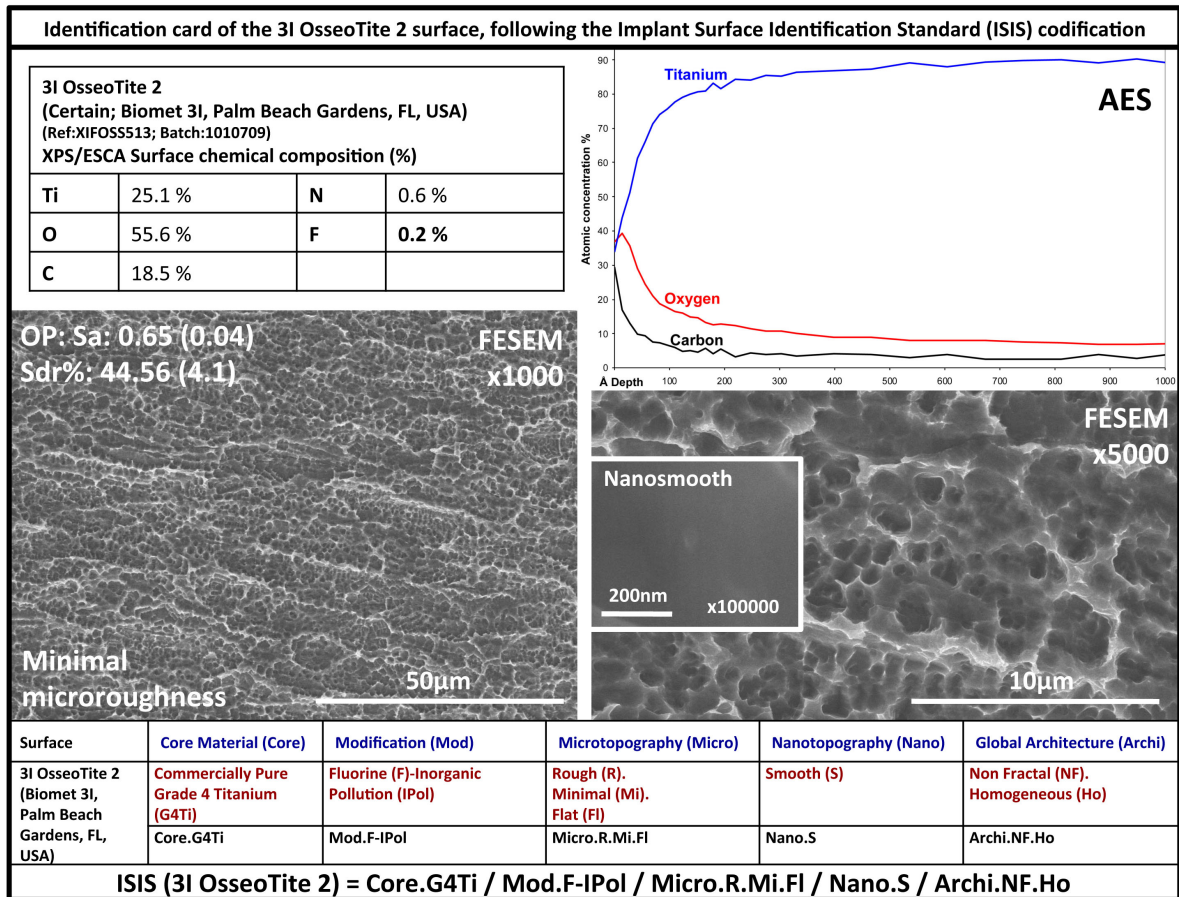


Figure 17. Identification Card of the 3I OsseoTite 2 surface.

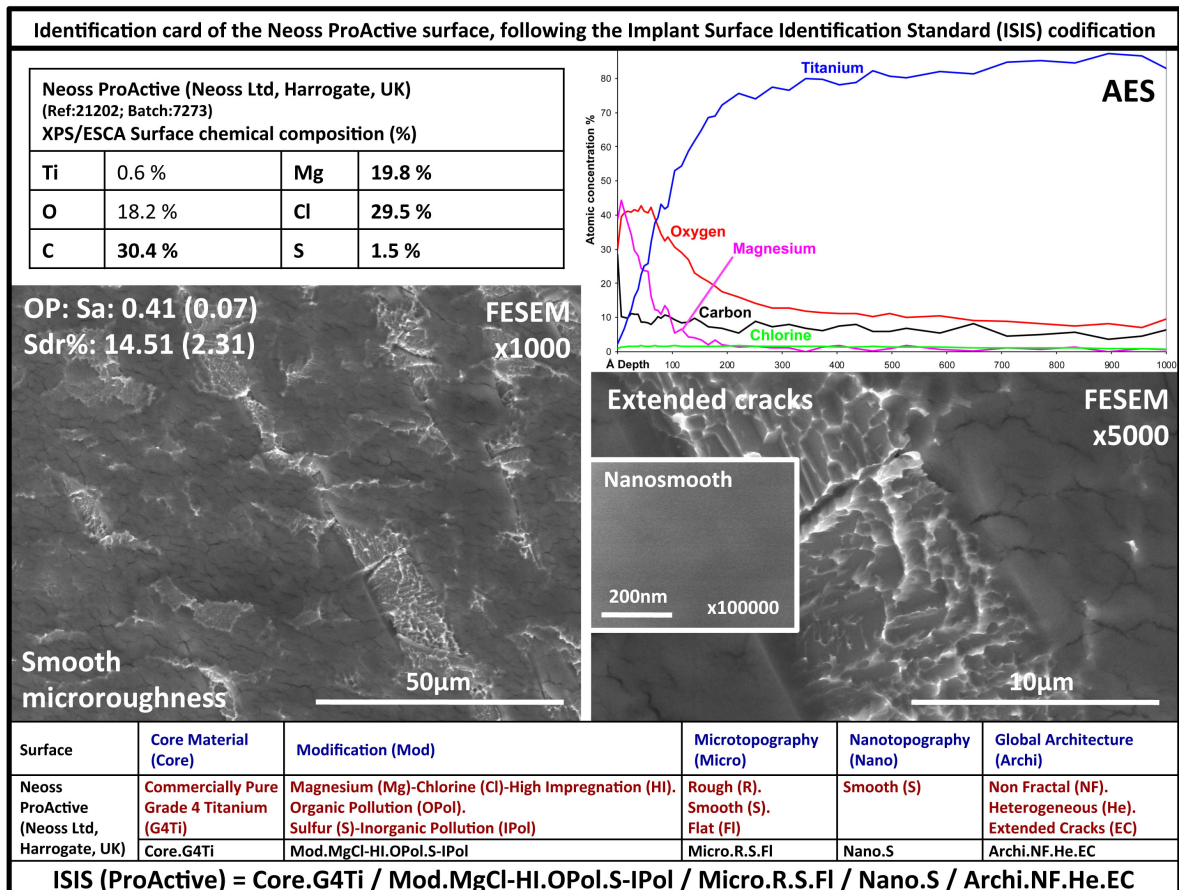


Figure 18. Identification Card of the Neoss ProActive surface.

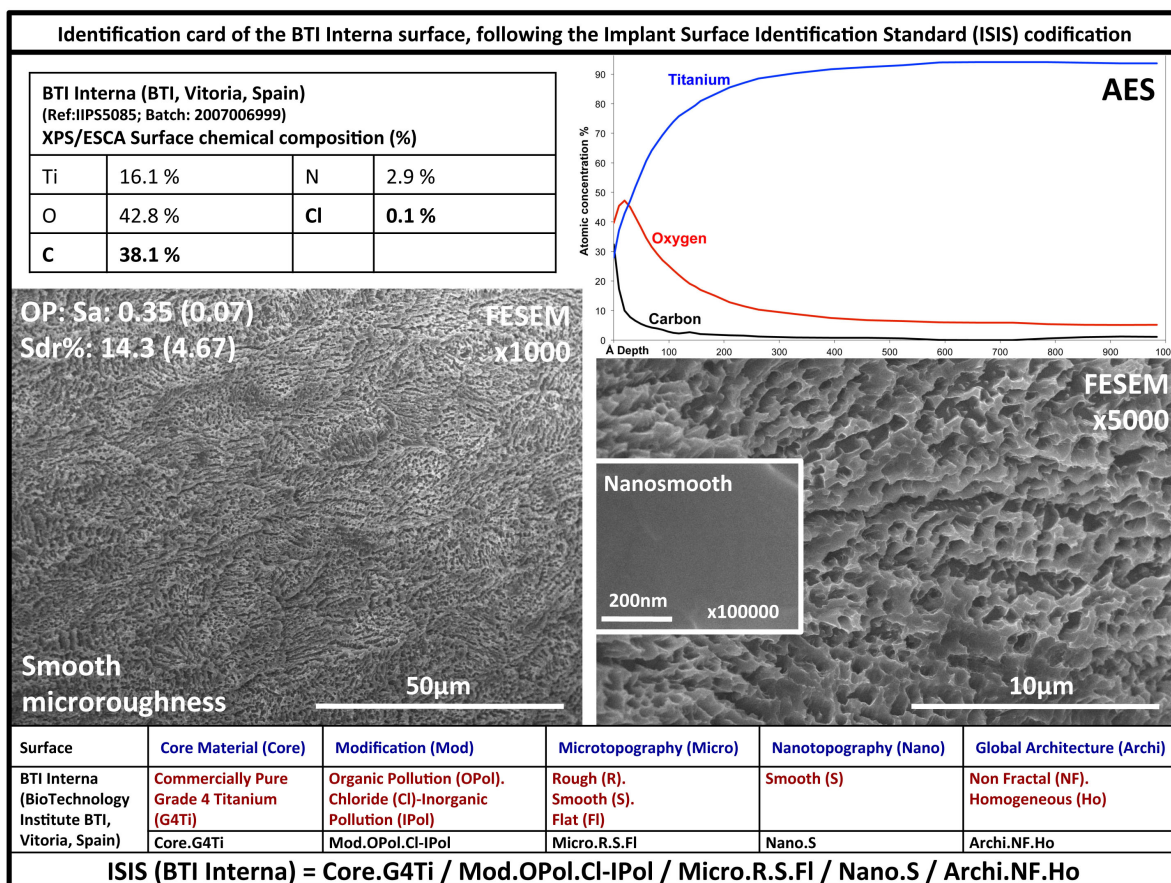


Figure 19. Identification Card of the BTI Interna surface.

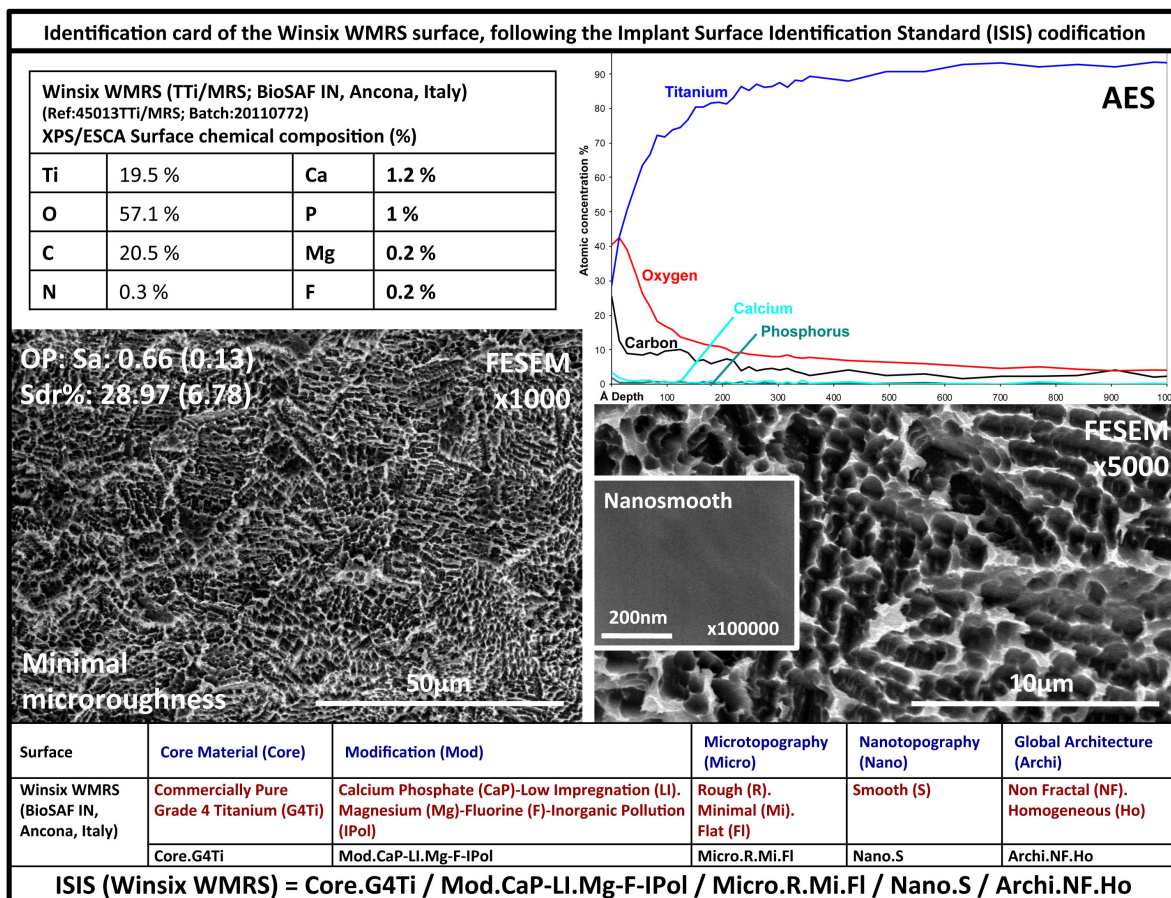


Figure 20. Identification Card of the Winsix WMRS surface.

Blossom Ossean (Intra-Lock, Boca Raton, Florida, USA; **Figure 5**) was a RBM-blasted/washed surface following a Subtractive Impregnation Micro/Nanotexturization (SIMN) unknown process. Blossom Ossean was an upgrade of Ossean, tailored for specific implant design. The surface was impregnated with low levels of calcium phosphate (CaP) and silicon (as titanium silicate), not visible with FE-SEM but homogeneous all over the surface. No pollution was detected. The microroughness was minimal, but close to the moderate level, and was covered with a nanoroughness all over the implant. The surface was homogeneous in chemistry and topography, and could be considered as fractal following our definition.

Osstem RBM (Osstem implant Co., Busan, Korea; **Figure 6**) was a RBM-blasted/washed surface. The surface was impregnated with low levels of calcium phosphate (CaP), not visible with FE-SEM but homogeneous all over the surface. Some inorganic pollution with silicon was also detected. The surface was minimally microrough, nanosmooth, and homogeneous all over the implant.

Blossom Ossean G23 ELI (Intra-Lock, Boca Raton, Florida, USA; **Figure 7**) was a RBM-blasted/washed surface following a Subtractive Impregnation Micro/Nanotexturization (SIMN) unknown process, on a grade 23 ELI (Extra Low Interstitials) titanium core. Blossom Ossean G23 ELI was a variation of the standard Blossom Ossean (grade 4 titanium) applied on grade 23 ELI titanium alloys (for smaller diameter implants particularly). The surface was impregnated with low levels of calcium phosphate (CaP) and silicon (as titanium silicate), not visible with FE-SEM but homogeneous all over the surface. No pollution was detected. The microroughness was minimal, but close to the moderate level, and was covered with a nanoroughness all over the implant. The surface was homogeneous in chemistry and topography, and could be considered as fractal following our definition. Except the difference in core material, this surface appeared exactly similar to the standard Blossom Ossean.

SBM Dual Blast body (Soluble Blast Media; Implant Direct LLC, Calabasas, CA, USA; **Figure 8**) was a RBM-blasted/washed surface on a grade 5 titanium core. This implant presented in fact 2 SBM surfaces: the body was more textured than the cervical area, what explained the name “Dual Blast”, and the cervical area was analyzed in the Group 4 (fifth part of this series of articles). The surface was impregnated with low levels of calcium phosphate (CaP), not visible with FE-SEM but homogeneous all over the surface. Some inorganic pollution with silicon was also detected. The surface was moderately microrough, nanosmooth, and homogeneous all over the implant.

MegaGen RBM (MegaGen Co., Seoul, Korea; **Figure 9**) was a RBM-blasted/washed surface. The surface was impregnated with low levels of calcium phosphate (CaP), not visible with FE-SEM but homogeneous all over the surface. Some inorganic pollution with silicon and sulfur was also detected. The surface was minimally microrough, nanosmooth, and homogeneous all over the implant.

DIO BioTite-M (DIO Corporation, Busan, Korea; **Figure 10**) was a RBM-blasted/washed surface. The surface was impregnated with residual levels of calcium phosphate (CaP), not visible with FE-SEM but homogeneous all over the surface. Significant inorganic pollutions with magnesium, silicon and particularly tungsten were also detected. The surface was minimally microrough, nanosmooth, and homogeneous all over the implant.

Blue Sky Bio RBM (Blue Sky Bio, Grayslake, IL, USA; **Figure 11**) was a RBM-blasted/washed surface on a grade 5 titanium core. The surface was impregnated with high levels of calcium phosphate (CaP), not visible with FE-SEM but homogeneous all over the surface. The surface was moderately microrough, nanosmooth, and homogeneous all over the implant.

Anthogyr BCP (Biphasic Calcium Phosphate; Anthogyr, Sallanches, France; **Figure 12**) was a RBM-blasted/washed surface on a grade 5 titanium core. The surface was impregnated with low levels of calcium phosphate (CaP), not visible with FE-SEM but homogeneous all over the surface. Some organic pollution (carbon overcoat) and some inorganic pollution with silicon were also detected. The surface was moderately microrough, nanosmooth, and homogeneous all over the implant.

Shinhung RBM+ (Shinhung Co., Seoul, Korea; **Figure 13**) was a RBM-blasted/washed surface. The surface was impregnated with residual levels of calcium phosphate (CaP), not visible with FE-SEM but homogeneous all over the surface. Some inorganic pollution with silicon was also detected. The surface was minimally microrough, nanosmooth, and homogeneous all over the implant.

Neobiotech CMI (Neobiotech Co., Seoul, Korea; **Figure 14**) was a RBM-blasted/washed surface. The surface was impregnated with low levels of calcium phosphate (CaP), not visible with FE-SEM but homogeneous all over the surface. Some inorganic pollution with silicon was also detected. The surface was minimally microrough, nanosmooth, and homogeneous all over the implant.

3.3. Other surfaces of the Group 2B

The 6 other surfaces of this group were produced through various surface subtractive processes, particularly dual acid-etching, TiO₂-blasting/acid-etching and others rare undefined process. Various forms of impregnation and/or pollutions were detected. All surfaces had in common to be microrough (with different degrees of roughness) and 5 were nanosmooth. Only 1 was nanorough, following a Subtractive Impregnation Micro/Nanotexturization (SIMN).

Osseospeed (AstraTech, Mölndal, Sweden; **Figure 15**) was produced through blasting with TiO₂ particles and etching with hydrofluoric acid, following a Subtractive Impregnation Micro/Nanotexturization (SIMN) unknown process. The surface was impregnated with residual levels of fluoride. No pollution was detected. The microroughness was moderate, and covered with a nanoroughness all over the implant. Some large TiO₂ residual blasting particles were impacted in the surface and presented a very smooth surface (at both micro- and nanoscale). For this reason, the surface could be considered heterogeneous.

3I OsseoTite (Biomet 3I, Palm Beach Gardens, FL, USA; **Figure 16**) was a dual acid-etched surface on a grade 5 titanium core. Some inorganic pollution with fluorine was detected. The surface was smooth at the microscale, smooth at the nanoscale and homogeneous all over the implant.

3I OsseoTite 2 (Biomet 3I, Palm Beach Gardens, FL, USA; **Figure 17**) was a dual acid-etched surface on a grade 4 titanium core. Some inorganic pollution with fluorine was detected. The surface was minimally microrough, nanosmooth, and homogeneous all over the implant. OsseoTite and OsseoTite 2 were in theory the same dual acid-etched process and surface, the only difference being the core material on which the acid-etching was applied; the surfaces were in fact a little bit different in microtopography, probably due to the different hardness between the 2 core materials during surface processing.

Neoss ProActive (Neoss Ltd, Harrogate, UK; **Figure 18**) was a blasted/etched and chemically altered surface using an unknown process (electrowetting). The surface was impregnated with high but heterogeneous levels of magnesium and chlorine all over the surface. Significant organic pollution (carbon overcoat) and some inorganic pollution with

sulfur were also detected. The surface was smooth at the microscale, smooth at the nanoscale, heterogeneous all over the implant and covered with many extended cracks (the reason of these cracks was unknown, but related to the surface processing).

BTI Interna (Biotechnology Institute, Vitoria, Spain; **Figure 19**) was a combined acid-etched surface. The surface appeared covered with a thick organic pollution (thick carbon species overcoat all over the implant). Some inorganic pollution with chlorine was also detected. The surface was smooth at the microscale (with typical small etching pits aspect and a low global height deviation amplitude), smooth at the nanoscale and homogeneous all over the implant.

Winsix WMRS (Winsix Micro-Rough Surface; BioSAF IN, Ancona, Italy; **Figure 20**) was a combined acid-etched surface. The surface was impregnated with low levels of calcium phosphate (CaP). Some inorganic pollution with magnesium and fluorine was also detected. The surface was minimally microrough (with typical small etching pits aspect and a low global height deviation amplitude), nanosmooth and homogeneous all over the implant.

4. Discussion

The RBM-type surfaces represented the second largest sub-group from all the implant surface technologies that were investigated in this study. This subtractive process is largely used in the industry [7]. All these products have in common some characteristics, such as a typical microrough morphology and the absence of significant nanofeatures. The variations of the process allow to carve different morphologies on the dental implant surface, particularly the degree of aggressiveness of the microroughness, but the general aspect of this subgroup is very typical and easy to recognize. The concept of the RBM-type surfaces is to promote a bone/implant biomechanical interlocking through the microroughness carved on the implant surfaces, but also to promote some bone/implant biochemical interlocking through the impregnation with CaP during the blasting process [1,12]. CaP impregnation is expected to serve as a promoter of biomineralization through ionic chelation and direct cell stimulation [10]. This type of surface is often associated with good clinical results [8], and it explains its frequent use in the industry.

In the RBM sub-group, two types of surfaces presented some significant differences with the general patterns of this sub-group. The first was Adin OsseoFix, where the RBM was not washed and removed at the end of process, leaving a large quantity of CaP blasting residues like a semi-coating all over the surface; this strategy was advocated to increase de CaP chemical modification (similar to a coated surface like Nanotite)[13], but this approach raises significant questions as the modification appeared heterogeneous and uncontrolled. The second types were the various forms of Intra-Lock Ossean which was the only RBM variation to present also a specific nanoroughness, related to specific processing and post-processing; this method is therefore a Subtractive Impregnation Micro/Nanotexturization (SIMN) process [10].

The CAE-type surfaces represented a quite small sub-group, mostly used by a limited number of companies [9]. All these surfaces presented a typical etched microrough morphology appearing flat and smooth at the microscale and the absence of significant nanofeatures. The low microroughness is expected to promote some bone/implant biomechanical interlocking and to reduce the risk of bacterial contaminations in the implant neck (in comparison to rougher and more aggressive surface morphologies), even if the link between smooth surfaces and low peri-implantitis risk was never proven and remained

theoretical [14]. No chemical modification for biochemical interlocking is advocated with this technology, except if a supplementary coating is applied on the surface (for example, this is how OsseoTite was transformed in the NanoTite surface, described in the Group 3)[13]. The typical etched morphology is often advocated to be more hydrophilic for blood and cell adhesion, but again this concept was not really supported, as the surface energy and hydrophilicity is first of all related to a micro-nanoroughness equilibrated combination. Practically, this kind of smooth morphology is often associated with lower bone/implant torque removal and reduced osseointegration strength [15,16], what may explain the relatively limited use of this technology nowadays.

Other surface technologies exist with other combinations of blasting and/or etching. One surface (Neoss) was difficult to identify and define accurately. The exact process was unclear, even if the company described it as a combination of blasting, etching and chemical treatment. Practically, after analysis, the Neoss surface appeared very smooth and flat (even more than a CAE surface) at the micro- and the nanoscale, presented heterogeneous high magnesium chlorine impregnation without modification of the TiO₂ layer thickness (no electrochemical modification), and a general very heterogeneous aspect with extended cracks.

Some other combinations of technologies lead to the development of Subtractive Impregnation Micro/Nanotexturization (SIMN), what is nowadays the main evolution and path of improvement of the subtractive technologies. One characteristic of most surfaces produced by subtraction evaluated in this article and the previous article (SLA-type, RBM, CAE) was that they did not present significant nanofeatures: all were nanosmooth [17]. The only nanotextured surfaces in this large family of subtractive processes were Astra Osseospeed [11] and the various forms of Intra-Lock Ossean [10]: they are the 2 main (and probably only) examples of Subtractive Impregnation Micro/Nanotexturization (SIMN) on the market and represent the latest evolution of the wide subtractive family [15,18].

Astra Osseospeed is famous and unique for its surface carving through blasting with TiO₂ particles and etching with hydrofluoric acid in specific conditions [4]. The fluorine impregnation was described as an efficient chemical modification [11,15], but many implant surfaces presented similar fluorine levels as pollutions. The design of its nanofeatures was unique and easy to recognize; no other surface presented similar patterns at the nanoscale [17]. This SIMN surface was however heterogeneous, because of the presence of many large TiO₂ particles as blasting residues impacted in the core material, which were very smooth at the micro and nanoscale. Moreover, the fluorine impregnation was not fully homogeneous, as it was a residue of etching.

Intra-Lock Ossean was produced through the combination of RBM technology with specific processing and post-processing [10]. The CaP impregnation was quite common with RBM surfaces, but Ossean presented a specific unique nanotexture all over the implant surface. The nanofeatures were different (more nanometric) than the texturization of Osseospeed (which was close to reach the micrometric scale), the difference was obvious during observation. This surface was moreover homogeneous, as there was no blasting residue, no pollution and the CaP low impregnation was homogeneous during the AES check. This led to the qualification of fractal surface, as the surface presented the same homogeneous aspect (one-dimension modification) at each micro, nano and chemical level [5]. This fractal concept is an interesting approach for surface science and controlled SIMN technology may lead to more fractal surfaces in the future.

In this study, we had the opportunity to analyze 3 versions of this surface. The early Ossean on Grade 4 Titanium already presented the typical CaP low impregnation and nanoroughness [10]. The latest version used on the Blossom implants (Blossom Ossean Grade 4 Titanium) presented the same CaP low impregnation and nanoroughness, but the company added some Si low impregnation (as silicate) to dope the osseointegration. Finally, we also analyzed the aspect of Ossean on Grade 23 titanium; this titanium alloy is used with some implant design or diameter to improve the biomechanical characteristics of the implants, and therefore the core material is harder and requires efforts to recalibrate all the chain of production to reach the same surface characteristics (this is the reason why most companies use only one type of titanium for all their implants). In this study, we observed that Ossean was exactly the same on Titanium grade 4 and Titanium grade 23, what was also the sign of an important effort of the company to reach such exact match. This notion illustrates that companies should always check that the surfaces they produce are really the same when they change the material or the design of the implant, even if it requires a lot of efforts.

The results published with these surfaces produced through Subtractive Impregnation Micro/Nanotexturization SIMN are often very positive [10,11,15], what may allow to suspect that the SIMN is a relevant way of evolution for the subtractive technologies.

In this large pool of RBM/DAE samples, many samples presented some inorganic pollution, mostly silicon (often associated to packaging contaminants); some unexpected elements (tungsten) were also detected in this group. In general, the RBM samples seem to present less contaminants than the SLA type surfaces, but anyway some improvements of the industrial production cleanliness are still needed. In this series of 20 surfaces, we detected 4 surfaces with significant organic pollution. These kinds of contaminants are often related to higher risk of early implant failure or peri-implantitis [14]. This result raises some significant concerns of public health policy concerning the control of the industrial products available on the market.

5. Conclusion

The RBM-type surfaces represent one of the most frequent surface technologies used in the dental implant industry, and are based mostly on the concept of bone/implant biomechanical interlocking through the implant microroughness and biochemical interlocking through the presence of CaP. Like SLA-type surfaces, RBM surfaces are often associated with good clinical results, and it is probably the reason of their frequent use. DAE-type surfaces presented a specific small microroughness and are not widely used nowadays in the industry. All these surfaces were smooth at the nanoscale. The frequent presence of inorganic or organic contaminants on these products revealed that some improvements are often needed to increase the industrial quality. Finally, the SIMN surfaces appeared as a natural evolution for the various subtractive technologies, as they allowed to reach a specific chemical modification, microtexture and nanotexture. At this time, only 2 companies were able to control and use this technology. It is expected that the SIMN approach will develop slowly in the coming years, with the improvement of industrial productions.

Disclosure of interests

Like most specialists in the implant surface field of research, the authors of this article are currently involved in experimental studies with various dental implant companies. This codification

article thus does not give qualitative opinions and is strictly founded on physical and chemical definitions, in order to avoid any subconscious conflict of interest. Moreover, the chemical values (XPS/AES) and the morphological data shown in the ID cards were double-checked by independent laboratories. This work has not been supported by grants from any commercial companies.

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Author Contributions

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References

- [1] Dohan Ehrenfest DM, Coelho PG, Kang BS, Sul YT, Albrektsson T. *Classification of osseointegrated implant surfaces: materials, chemistry and topography*. **Trends Biotechnol.** 2010;28(4):198-206.
- [2] Dohan Ehrenfest DM, Rutkowski JL. *Evolution of the dental implant market: an African tale revisited*. **J Oral Implantol.** 2012;38(3):201-2.
- [3] Kang BS, Sul YT, Oh SJ, Lee HJ, Albrektsson T. *XPS, AES and SEM analysis of recent dental implants*. **Acta Biomater.** 2009;5(6):2222-9.
- [4] Dohan Ehrenfest DM, Vazquez L, Park YJ, Sammartino G, Bernard JP. *Identification card and codification of the chemical and morphological characteristics of 14 dental implant surfaces*. **J Oral Implantol.** 2011;37(5):525-42.
- [5] Dohan Ehrenfest DM. *Fractal patterns applied to implant surface: definitions and perspectives*. **J Oral Implantol.** 2011;37(5):506-9.
- [6] Dohan Ehrenfest DM, Kang BS, Sammartino G, Shibli JA, Wang HL, Zou DR, Bernard JP. *Guidelines for the publication of articles related to implant surfaces and design from the POSEIDO: a standard for surface characterization*. **POSEIDO.** 2013;1(1):7-15.
- [7] Coelho PG, Granjeiro JM, Romanos GE, Suzuki M, Silva NR, Cardaropoli G, Thompson VP, Lemons JE. *Basic research methods and current trends of dental implant surfaces*. **J Biomed Mater Res B Appl Biomater.** 2009;88(2):579-96.
- [8] Ormianer Z, Piek D, Livne S, Lavi D, Zafrir G, Palti A, Harel N. *Retrospective clinical evaluation of tapered implants: 10-year follow-up of delayed and immediate placement of maxillary implants*. **Implant Dent.** 2012;21(4):350-6.
- [9] Testori T, Del Fabbro M, Feldman S, Vincenzi G, Sullivan D, Rossi R, Jr., Anitua E, Bianchi F, Francetti L, Weinstein RL. *A multicenter prospective evaluation of 2-months loaded Osseotite implants placed in the posterior jaws: 3-year follow-up results*. **Clin Oral Implants Res.** 2002;13(2):154-61.
- [10] Bucci-Sabattini V, Cassinelli C, Coelho PG, Minnici A, Trani A, Dohan Ehrenfest DM. *Effect of titanium implant surface nanoroughness and calcium phosphate low impregnation on bone cell activity in vitro*. **Oral Surg Oral Med Oral Pathol Oral Radiol Endod.** 2010;109(2):217-24.
- [11] Monjo M, Lamolle SF, Lyngstadaas SP, Ronold HJ, Ellingsen JE. *In vivo expression of osteogenic markers and bone mineral density at the surface of fluoride-modified titanium implants*. **Biomaterials.** 2008;29(28):3771-80.
- [12] Sul YT, Kang BS, Johansson C, Um HS, Park CJ, Albrektsson T. *The roles of surface chemistry and topography in the strength and rate of osseointegration of titanium implants in bone*. **J Biomed Mater Res A.** 2009;89(4):942-50.
- [13] Abrahamsson I, Linder E, Larsson L, Berglundh T. *Deposition of nanometer scaled calcium-phosphate crystals to implants with a dual acid-etched surface does not improve early tissue integration*. **Clin Oral Implants Res.** 2013;24(1):57-62.

- [14] Mouhyi J, Dohan Ehrenfest DM, Albrektsson T. *The peri-implantitis: implant surfaces, microstructure, and physicochemical aspects*. **Clin Implant Dent Relat Res**. 2012;14(2):170-83.
- [15] Coelho PG, Granato R, Marin C, Bonfante EA, Freire JN, Janal MN, Gil JN, Suzuki M. *Biomechanical evaluation of endosseous implants at early implantation times: a study in dogs*. **J Oral Maxillofac Surg**. 2010;68(7):1667-75.
- [16] Sul YT, Johansson C, Albrektsson T. *Which surface properties enhance bone response to implants? Comparison of oxidized magnesium, TiUnite, and Osseotite implant surfaces*. **Int J Prosthodont**. 2006;19(4):319-28.
- [17] Shibli JA, Dohan Ehrenfest DM. *In dental implant surfaces, NanoWar has begun... but NanoQuest is still at stake!* **POSEIDO**. 2013;1(3):131-40.
- [18] Mendonca G, Mendonca DB, Aragao FJ, Cooper LF. *Advancing dental implant surface technology--from micron- to nanotopography*. **Biomaterials**. 2008;29(28):3822-35.

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