

## Research article

**Identification card and codification of the chemical and morphological characteristics of 62 dental implant surfaces. Part 5: chemically coated surfaces (Group 3, coating) and implant collar surfaces (Group 4, collar)**

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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 fifth part, coated surfaces and some collar surfaces were investigated.

**Materials and Methods.** Sixteen different implant surfaces were characterized: NanoTite (Biomet 3I, Palm Beach Gardens, FL, USA), SLActive (ITI Straumann, Basel, Switzerland), Roxolid SLActive (ITI Straumann, Basel, Switzerland), Xpeed (MegaGen Co., Seoul, Korea), Xpeed Plus (MegaGen Co., Seoul, Korea), Inicell (Thommen, Waldenburg, Switzerland), Integra-CP/NanoTite (Bicon, Boston, MA, USA), Dentis Haptite (Dentis, Daegu, Korea), Legacy 2 HA (Implant Direct LLC, Calabasas, CA, USA), Biohorizons HA (Biohorizons, Birmingham, AL, USA), Osstem HA (Osstem implant Co., Busan, Korea), DIO BioTite-H (DIO Co., Busan, Korea), Laser-Lok collar (Biohorizons, Birmingham, AL, USA), SBM collar (Implant Direct, Calabasas, CA, USA), Ossean collar (Intra-Lock, Boca Raton, Florida, USA), Kohno DES ZirTi (Sweden & Martina, Due Carrare, 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 16 different surfaces of this group, 5 were based on a commercially pure titanium (grade 4), 4 on a titanium-aluminium alloy (grade 5 or 23), 1 on a titanium-zirconium alloy, 3 on hydroxyapatite, 1 on brushite and 2 on a calcium phosphate core. 13 surfaces presented different forms of chemical impregnation or discontinuous coating of the core material. 15 surfaces presented different degrees of inorganic pollutions, and 1 presented also some organic pollution overcoat. Only 1 surface presented no pollution (Ossean collar). From a morphological standpoint, 1 surface was micropatterned (laser patterning) and 15 microrough, with different microtopographical aspects and values. 8 surfaces were smooth on the nanoscale, and therefore presented no significant and repetitive nanostructures. Eight surfaces were nanomodified: 2 implants were nanorough (Haptite and Ossean collar) and 6 were covered with nanoparticles (CaP, NaCl or Ca nanocrystals deposition: NanoTite, SLActive and Xpeed). Hydroxyapatite and brushite coated surfaces were heterogeneous and covered with extended cracks all over the surface. Only 6 surfaces were homogeneous and 10 were heterogeneous. Only one surface (Ossean collar) was 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. Coated surfaces had very specific morphological characteristics depending on the type of coating (nanocrystals heterogeneous deposition, or heterogeneous maximal microroughness with extended cracks for example). 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. The development of new surfaces for the implant cervical area is also an important clinical paradigm users should be aware about. Finally, the diversity of the surfaces analyzed in this study illustrated that the ISIS system could be an interesting basis for the development of a clear and simple ISO standard for dental implant surfaces and other implantable devices.

**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 [1,2]. The surface characteristics are often advertised by the dental implant companies in order to promote their products, but most data remain very commercial and without certified evaluation and disclosure of the surfaces characteristics [1,3]. 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.

This card describes the surface chemical composition and morphological characteristics of each surface [4,5]. 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, 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 third category of methods (arbitrarily termed Group 3) to create a dental implant surface is to cover the core material with a chemical coating. Two types of coating can be clearly distinguished. The first type (Group 3A) regroups titanium-based surface with a nanometric coating (often discontinuous), where a titanium surface is prepared through a subtractive classical technique (Sand-blasted and Acid-etched (SLA-type), Dual Acid-Etched (DAE) or blasted with Resorbable Blasting Media (RBM)) and finally coated with a thin final chemical preparation (nanometric, less than 100 nm thick). The most frequent method nowadays is to place a SLA-type implant in a NaCl or NaOH solution in order to provoke the formation of Na-based temporary nanocrystals on the surface [4,7]; this approach was developed for Straumann SLActive surfaces, Thommen Inicell and was then frequently copied by other companies. Other methods used the deposition of a thin layer of calcium phosphate CaP on a microrough surface, to create Ca or CaP nanocrystals all over the titanium surface (the most famous example in this type being the 3I NanoTite surface)[4,8]. The concept of this Group 3A is to add some chemical modification and nanofeatures to a classical Group 2 surface (subtractive), and is nowadays more and more fashionable (particularly through the copies of SLActive).

The second type (Group 3B) regroups implants covered with a micrometric thick layer of hydroxyapatite (mostly Plasma-Sprayed Hydroxyapatite PSHA)[9] or other forms of CaP (ion-beam deposited layers, brushite coating)[10], without detectable titanium at the surface level. In this type, the core material of the surface is no more titanium but a combination of CaP. The osseointegration mechanisms are different as the bone is never in direct contact with the titanium oxide layer of the implant, and the whole integration process goes through CaP mineral interactions [1]. This kind of surface was fashionable some years ago, but is nowadays abandoned in Europe and relatively rare in America and Asia, due to some failures in the past years related to the delamination of the HA coatings. It is however still advocated in low quality bone (the HA being expected to stimulate the bone apposition)[9] and used when associated with specific implant concepts (such as the plateau root implant from Bicon)[10].

The fourth category of dental implant surfaces (arbitrarily termed Group 4) regroups the various methods developed to create a surface in the cervical collar area of some implants. These surfaces have the particularity to be designed to promote peri-implant cervical bone stability and the sealing of the implant/bone/gingiva interface, and to control the risk of bacterial contamination [11]. Most companies use the same surface all over the implants, while a minority of companies proposed specific surface designs for the collar area. The development of this kind of specific collar surfaces remains still limited and there is no consensus on the concepts that may support the characteristics of collar surfaces. Therefore companies are proposing various solutions, following their own rationale: micropatterns to improve the collar sealing [12] or on the contrary smoother microroughness to avoid bacterial colonization [13], chemical modification for mineral chelation or not, and sometimes nanofeatures [14]. These collar surfaces are based on the classical 3 groups of

surfaces (modification of titanium metallurgy, subtraction through blasting and/or etching, coating), and they represent an interesting and significant path of research in the field for the coming years.

In this fifth part, the chemical and morphological characteristics of 12 implant surfaces (available on the market) from the third group and 4 from the fourth group were investigated and described through a simple and clear identification (ID) card for each surface, following the ISIS system terminology and classification. The third group gathered all surfaces produced through chemical coating, while the fourth group gathers all surfaces designed specifically for the collar cervical area of the implant (to promote a better stability of the peri-implant bone and gingival attachment).

## 2. Materials and Methods

### 2.1. Samples

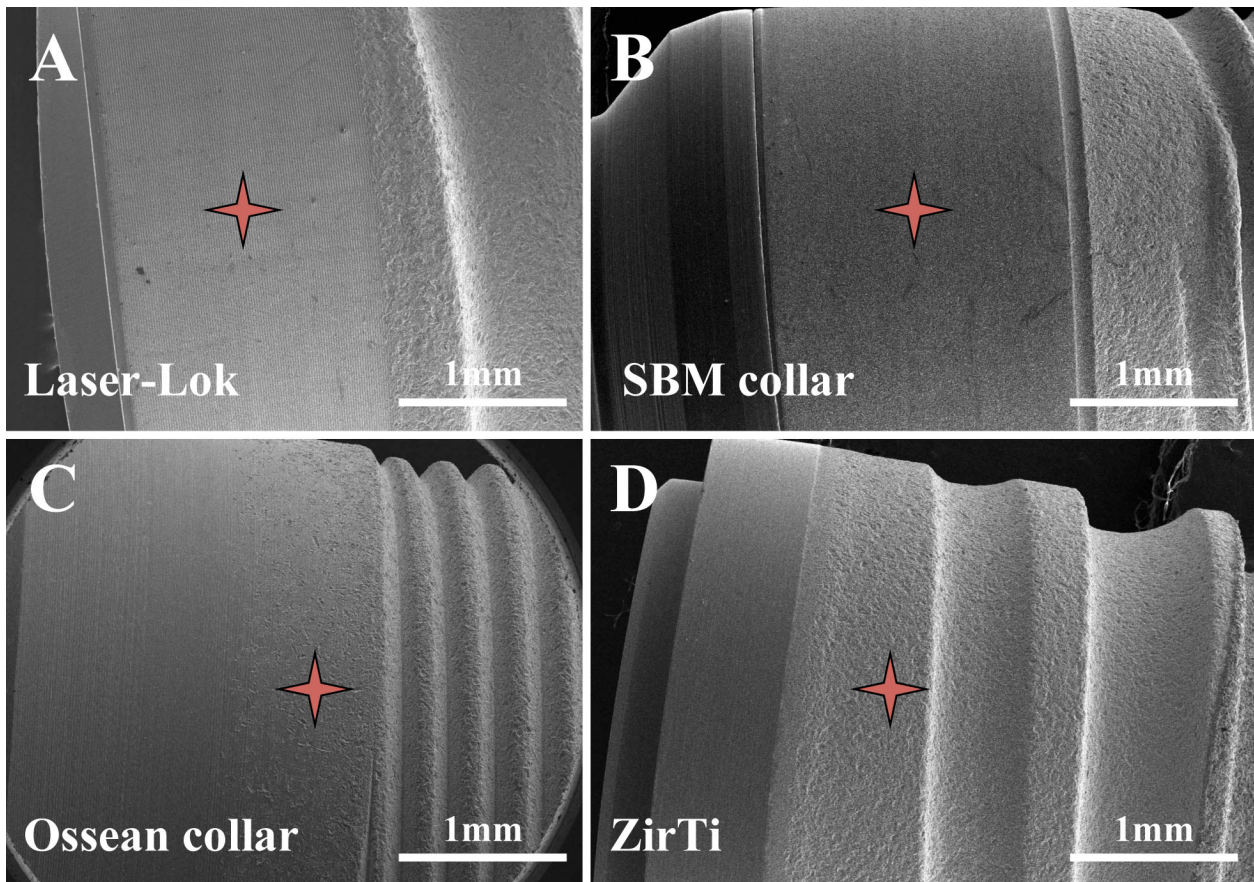
Sixteen different implant surfaces have been investigated. Six samples were titanium-based surfaces with a nanometric coating (Group 3A): NanoTite (Biomet 3I, Palm Beach Gardens, FL, USA), SLActive (ITI Straumann, Basel, Switzerland), Roxolid SLActive (ITI Straumann, Basel, Switzerland), Xpeed (MegaGen Co., Seoul, Korea), Xpeed Plus (MegaGen Co., Seoul, Korea), Inicell (Thommen, Waldenburg, Switzerland).

Six samples were implants covered with a micrometric thick layer of hydroxyapatite or other forms of CaP, without detectable titanium at the surface level (Group 3B): Integra-CP/NanoTite (Bicon, Boston, MA, USA), Dentis Haptite (Dentis, Daegu, Korea), Legacy 2 HA (Implant Direct LLC, Calabasas, CA, USA), Biohorizons HA (Biohorizons, Birmingham, AL, USA), Osstem HA (Osstem implant Co., Busan, Korea), DIO BioTite-H (DIO Co., Busan, Korea).

Finally, four samples were collar surfaces (**Figure 1**) specifically designed for the interface with the peri-implant cervical tissues (Group 4): Laser-Lok collar (Biohorizons, Birmingham, AL, USA), SBM collar (Implant Direct, Calabasas, CA, USA), Ossean collar (Intra-Lock, Boca Raton, Florida, USA), Kohno DES ZirTi (Sweden & Martina, Due Carrare, Italy).

Three samples were used per implant system, and their reference and batch were reported in their respective ID card. Most 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 implants that were offered by the company.





**Figure 1. Scanning electron microphotograph (SEM) of the general overview of the implant cervical areas presenting specific surface designs (magnification x30).** The star in red is marking the center of the collar area that was analyzed. **(A)** Laser-Lok collar surface (Biohorizons, Birmingham, AL, USA). The collar presented regular peripheral rail micropatterns, while the body of the implant was covered with a RBT (Resorbable Blast Textured) surface. The line of separation between surfaces was easy to recognize. **(B)** SBM collar surface (Implant Direct, Calabasas, CA, USA). The collar presented a much smoother aspect than the body (covered with a rougher SBM surface). A microchannel carved on the implant macrodesign separated the 2 surface areas. **(C)** Ossean collar surface (Intra-Lock, Boca Raton, Florida, USA). The body of the implant was covered with a classical Ossean surface, and it changed into the specific Ossean collar surface after the last cervical microthreads. The line of separation was not very visible, as the 2 surfaces presented a relatively close macro-morphology. **(D)** Kohno DES ZirTi surface (Sweden & Martina, Due Carrare, Italy). The implant DES (Dual Engineered Surface) was covered with a TPS surface on 2/3 of the implant body (appearing in dark grey), and the last cervical 1/3 was covered with a much smoother ZirTi surface (appearing in light grey). The line of separation was very visible, due to the very different nature of both surfaces.

## 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  $\text{AlK}\alpha$  1486.6eV, acceptance angle  $\pm 23^\circ$ , take-off angle  $45^\circ$ , charge correction C1s 284.8 eV), on a  $100\mu\text{m}$  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  $30^\circ$  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  $\text{TiO}_2$ : 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.

In the cases of implants coated with thick layers of Calcium Phosphates or various forms of Hydroxyapatite (PSHA Plasma-Sprayed Hydroxyapatite, Brushite, etc.), the use of AES analysis was not suitable. When the AES electrons are hitting the surfaces coated with CaP, it provokes a strong charging effect that alters very significantly the results. The same problem appears with classic forms of SEM (Scanning Electron Microscope), and only the use of FE-SEM (Field Emission-SEM) allows to avoid this physical phenomenon. For this reason, the 5 implant references presenting this kind of surfaces (Dentis Haptite, Implant Direct HA, Biohorizons HA, Osstem HA, DIO BioTite-H) were treated separately following another protocol. The in-depth analysis of the chemical composition of the external surface layer was performed 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  $\text{AlK}\alpha$  1486.6eV, acceptance angle  $\pm 23^\circ$ , take-off angle  $45^\circ$ ), on a  $100\mu\text{m}$  diameter analysis area located in the middle of the cutting edge flat area (or an equivalent flat part, depending on the implant macrodesign) of each sample. The in-depth chemical profile was established down to 100nm, using sputtering cycles with a 1keV Ar+ source (Ar+ etching rate: 1.01nm/min). Two in-depth profiles were established per sample. The analysis area with XPS was much larger than with AES (technical limit), what did not allow a very accurate punctual homogeneity check, but the 2 spots were precisely located in 2 different but close areas of the surface microtopography.

## 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 50x50µm 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 16 different surfaces of this group, 5 were based on a commercially pure titanium (grade 4), 4 on a titanium-aluminium alloy (grade 5 or 23), 1 on a titanium-zirconium alloy, 3 on hydroxyapatite, 1 on brushite and 2 on a calcium phosphate core. 13 surfaces presented different forms of chemical impregnation or discontinuous coating of the core material. 15 surfaces presented different degrees of

inorganic pollutions, and 1 presented also some organic pollution overcoat. Only 1 surface presented no pollution (Ossean collar).

From a morphological standpoint, 1 surface was micropatterned (laser patterning) and 15 microrough, with different microtopographical aspects and values. 8 surfaces were smooth on the nanoscale, and therefore presented no significant and repetitive nanostructures. Eight surfaces were nanomodified: 2 implants were nanorough (Haptite and Ossean collar) and 6 were covered with nanoparticles (CaP, NaCl or Ca nanocrystals deposition: NanoTite, SLActive and Xpeed). Hydroxyapatite and brushite coated surfaces were heterogeneous and covered with extended cracks all over the surface. Only 6 surfaces were homogeneous and 10 were heterogeneous. Only one surface (Ossean collar) was 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. Titanium surfaces with a nanometric chemical coating (Group 3A)

The 6 surfaces of this group were all prepared following a standard subtractive process (dual acid-etching, sand-blasting/acid-etching, RBM-blasting) on a titanium core and covered with a final nanometric chemical coating as main chemical modification. Inorganic pollutions were detected in all samples. All tested surfaces had in common to be microrough (with different degrees of roughness), discontinuously coated with crystalline nanoparticles of CaP, NaCl or Ca, and heterogeneous in chemistry and nanotexture all over the implant.

3I NanoTite (Biomet 3I, Palm Beach Gardens, FL, USA; **Figure 2**) was a dual acid-etched surface on a grade 5 titanium core, with a final discontinuous coating with CaP particles (discrete crystalline deposition - DCD). Some inorganic pollutions with fluoride and sulfur were also detected. The microroughness was smooth and flat, and covered with CaP nanoparticles creating a significant texture on the nanoscale. The size of the CaP particles could however vary a lot, and many microparticles or CaP aggregates were randomly found on the surface. For this reason, the surface could be considered heterogeneous.

SLActive (Sand-blasted, Large-grit, Acid-etched Active; ITI Straumann, Basel, Switzerland; **Figure 3**) was a sand-blasted/acid-etched surface with a final immersion in a sodium chloride (NaCl) solution. The surface was therefore coated with NaCl as main chemical modification. Traces of other elements were also detected as inorganic pollution (fluoride, potassium, calcium and phosphate). The microtopography was moderately rough, and rugged. When the implant was outside its box, the solution dried quickly on the surface and created NaCl aggregates all over the implant; the surface was covered with a discontinuous coating of NaCl nanocrystals. However, the morphology of this coating was very heterogeneous.

Roxolid SLActive (ITI Straumann, Basel, Switzerland; **Figure 4**) was a sand-blasted/acid-etched surface on a specific titanium-zirconium alloy (trademarked Roxolid, and designed to increase screw implant mechanical strength, like other titanium alloys such as grade 5 or grade 23, particularly for small diameter implants), with a final immersion in a sodium chloride (NaCl) solution. The surface was therefore coated with NaCl as main chemical modification. Inorganic pollutions with aluminium, fluorine and silicon were also detected. The microtopography was moderately rough, and rugged. When the implant was outside its box, the solution dried quickly on the surface and created NaCl aggregates all over the implant; the surface was covered with a discontinuous coating of NaCl nanocrystals. However, the morphology of this coating was very heterogeneous. This surface



microtopography appeared very similar to the standard SLActive made on grade 4 titanium, only the core materials and pollutions were different.

Xpeed (MegaGen Co., Seoul, Korea; **Figure 5**) was produced through blasting with a Resorbable Blasting Media (RBM) followed by washing of the particles (RBM-blasted/washed surface), with a final discontinuous coating with calcium Ca particles (alkaline calcium deposition). The surface was therefore coated with calcium as main chemical modification. Inorganic pollutions with iron, silicon and sulfur were also detected. The microtopography was moderately rough, flattened out and covered with a discontinuous coating of Ca nanocrystals. However, the morphology and chemistry of this coating could vary significantly all over the surface; for this reason, the surface could be considered heterogeneous.

Xpeed Plus (MegaGen Co., Seoul, Korea; **Figure 6**) was a sand-blasted/acid-etched surface with a final discontinuous coating with calcium Ca particles (alkaline calcium deposition). The surface was therefore coated with calcium as main chemical modification. Inorganic pollutions with iron, silicon, sulfur and phosphorus were also detected. The microtopography was moderately rough, rugged and covered with a discontinuous coating of Ca nanocrystals. However, the morphology and chemistry of this coating could vary significantly all over the surface; for this reason, the surface could be considered heterogeneous.

Inicell (Thommen, Waldenburg, Switzerland; **Figure 7**) was a sand-blasted/acid-etched surface with a final immersion in a sodium hydroxide (NaOH) solution. The implant was stored in a dry tube, and the final surface was produced by the conditioning process where a cartridge with 0.05M NaOH was pushed in the implant compartment and the conditioning liquid was left in contact a few minutes with the implant (Apliquiq system). The surface was coated with Na and Cl as main chemical modification. Inorganic pollutions with silicon, sulfur, fluorine and magnesium were also detected. The microtopography was moderately rough. After application and removal of the conditioning solution, the solution dried quickly on the surface and created Na aggregates all over the implant; the surface was covered with a discontinuous coating of Na or NaCl nanocrystals. However, the morphology and chemistry of this coating could vary significantly all over the surface; for this reason, the surface could be considered heterogeneous.

### 3.3. Surfaces with a thick coating of HA or CaP (Group 3B)

The 6 implants of this group were all titanium screws covered with a micrometric thick coating of hydroxyapatite or other forms of calcium phosphates, without detectable titanium at the surface level (Group 3B), so that the coating material could be considered as the core material of the surface itself. Some impregnation with magnesium was frequent (4 samples). Inorganic pollutions were detected in all samples. All surfaces had in common to be microrough (with different degrees of roughness) and 5 were nanosmooth (only one presented some significant nanotexture). The 4 surfaces with the thicker coating (plasma-sprayed hydroxyapatite and brushite) all presented a maximal, rugged extra and heterogeneous microroughness and large extended cracks all over the surface.

Integra-CP, previously known as Bicon NanoTite (Bicon, Boston, MA, USA; **Figure 8**), was a blasted/etched surface with a final coating using calcium phosphate Ion-Beam Assisted Deposition (IBAD). The CaP coating was thicker than 100nm, and CaP was therefore the core material of this surface. Inorganic pollutions with fluoride and sulfur were also



detected. The surface was minimally microrough, nanosmooth, and homogeneous all over the implant.

Dentis Haptite (Dentis, Daegu, Korea; **Figure 9**) was produced through a specific unknown blasting process, with a Resorbable Blasting Media (RBM) that remained adsorbed and engraved in the titanium core material and covered it completely with a thick CaP coating. The surface was coated with a CaP layer thicker than 100nm (1-2µm thick), and CaP was therefore the core material of this surface. A low impregnation with magnesium and some inorganic pollution with fluorine were also detected. The surface was minimally microrough, nanorough, and homogeneous all over the implant.

Legacy 2 HA (Implant Direct LLC, Calabasas, CA, USA; **Figure 10**) was a plasma-sprayed hydroxyapatite (PSHA) surface. The implant body was coated with a continuous micrometric thick layer of hydroxyapatite HA (often 50 µm thick), and HA was therefore the core material of this surface. A low impregnation with magnesium and some inorganic pollution with fluorine and silicon were also detected. PSHA surfaces had important common morphological characteristics: the microroughness was maximal, rugged extra, heterogeneous, smooth on the nanoscale, and covered with many extended cracks (related to the cooling of the plasma-sprayed hydroxyapatite).

Biohorizons HA (Biohorizons, Birmingham, AL, USA; **Figure 11**) was a plasma-sprayed hydroxyapatite (PSHA) surface. The implant body was coated with a continuous micrometric thick layer of hydroxyapatite HA (often 50 µm thick), and HA was therefore the core material of this surface. A high impregnation with magnesium and some inorganic pollution with fluorine were also detected. PSHA surfaces had important common morphological characteristics: the microroughness was maximal, rugged extra, heterogeneous, smooth on the nanoscale, and covered with many extended cracks (related to the cooling of the plasma-sprayed hydroxyapatite).

Osstem HA (Osstem implant Co., Busan, Korea; **Figure 12**) was a plasma-sprayed hydroxyapatite (PSHA) surface. The implant body was coated with a continuous micrometric thick layer of hydroxyapatite HA (often 50 µm thick), and HA was therefore the core material of this surface. A low impregnation with magnesium and some inorganic pollution with fluorine and silicon were also detected. PSHA surfaces had important common morphological characteristics: the microroughness was maximal, rugged extra, heterogeneous, smooth on the nanoscale, and covered with many extended cracks (related to the cooling of the plasma-sprayed hydroxyapatite).

DIO BioTite-H (DIO Co., Busan, Korea; **Figure 13**) was a surface coated with a thick layer of brushite, a calcium phosphate mineral (often considered as the precursor of apatite). The use of brushite was very rare and it presented a specific and fragile crystalline flower morphology. The brushite thick layer was therefore the core material of this surface. Some inorganic pollutions with magnesium and fluorine were also detected. This brushite surface had important common morphological characteristics with PSHA surfaces: the microroughness was maximal, rugged extra, heterogeneous, smooth on the nanoscale, and covered with many extended cracks (related to the coating process).

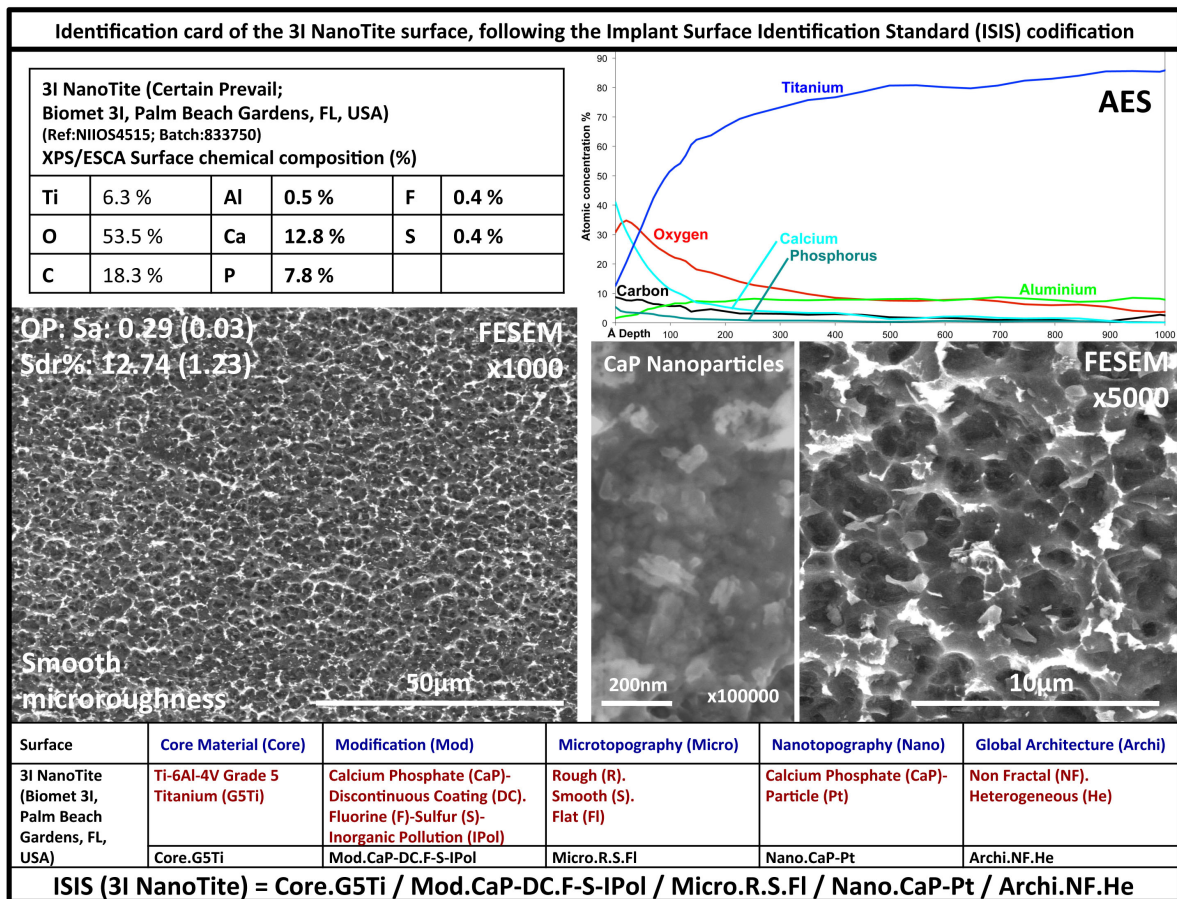


Figure 2. Identification Card of the 3I NanoTite surface.

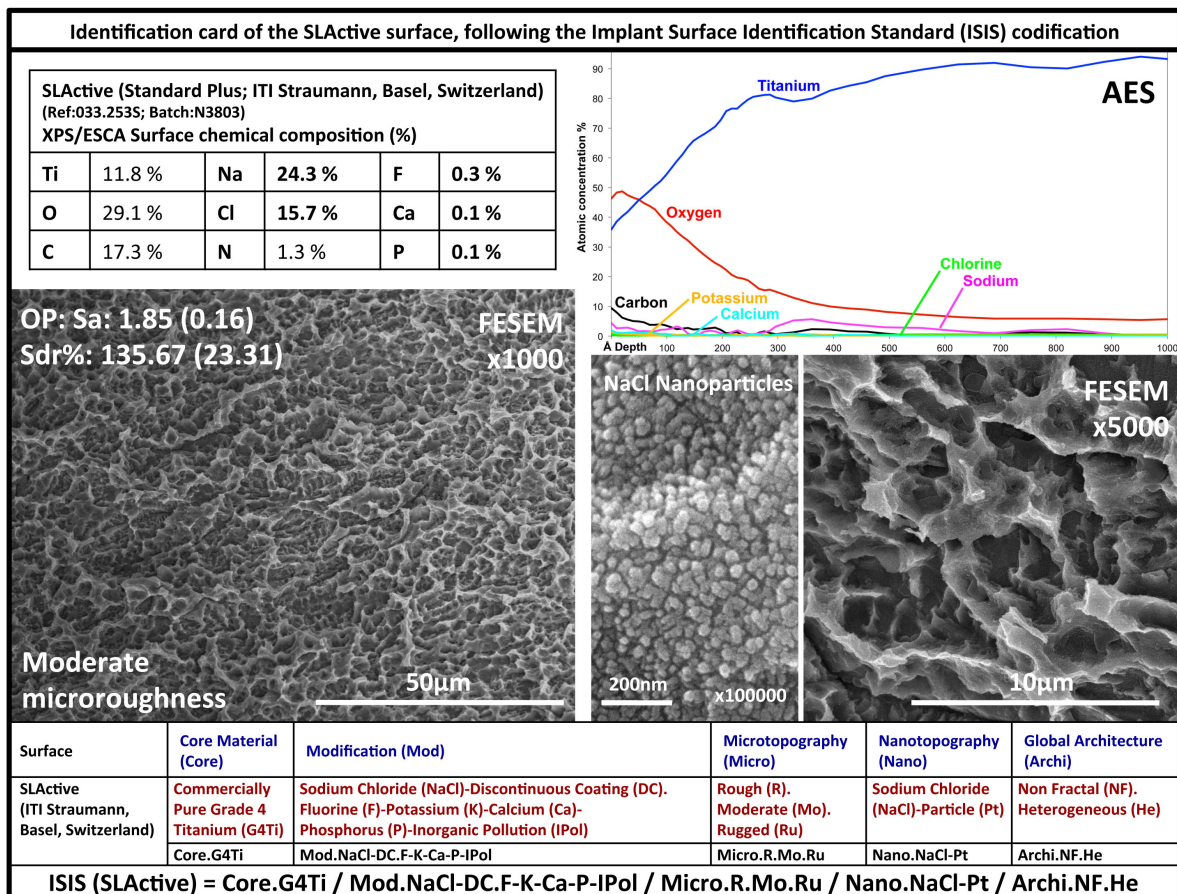
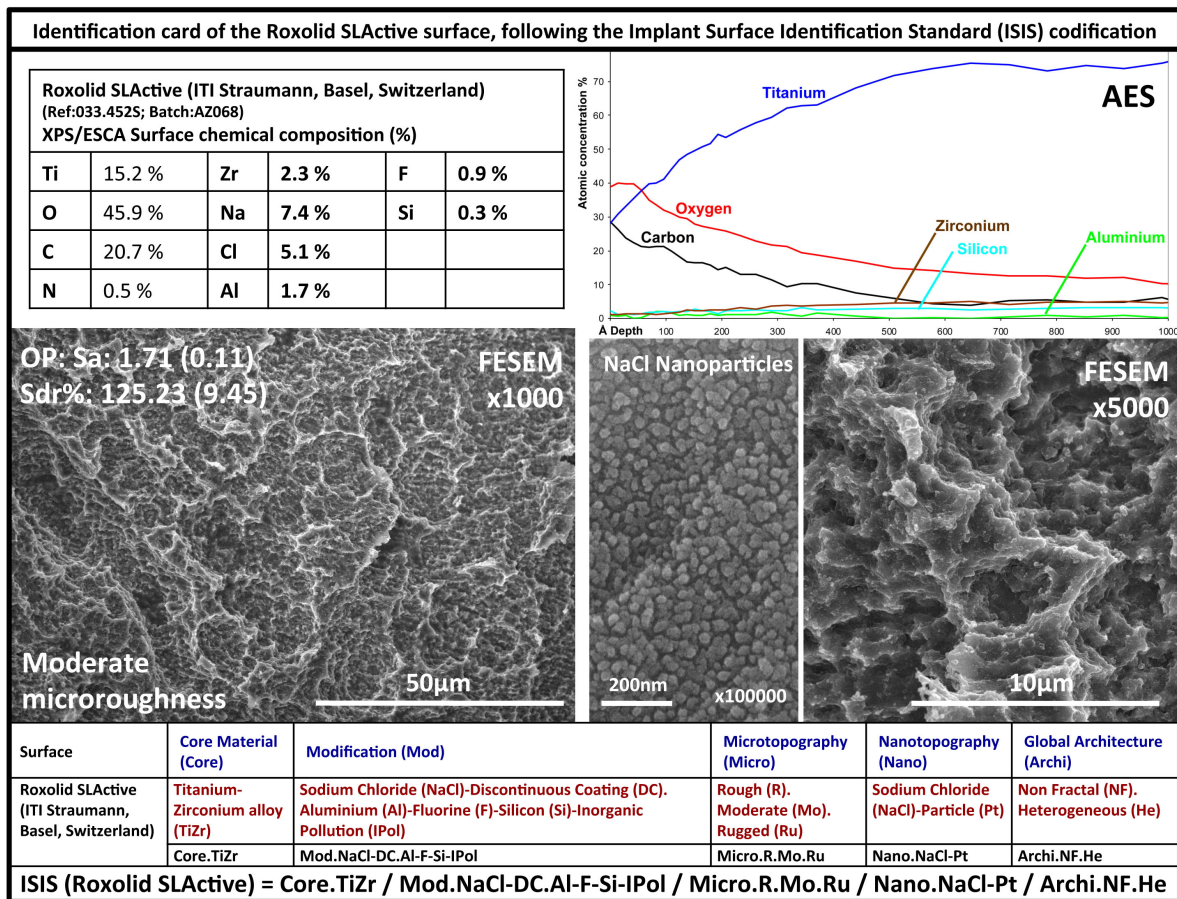
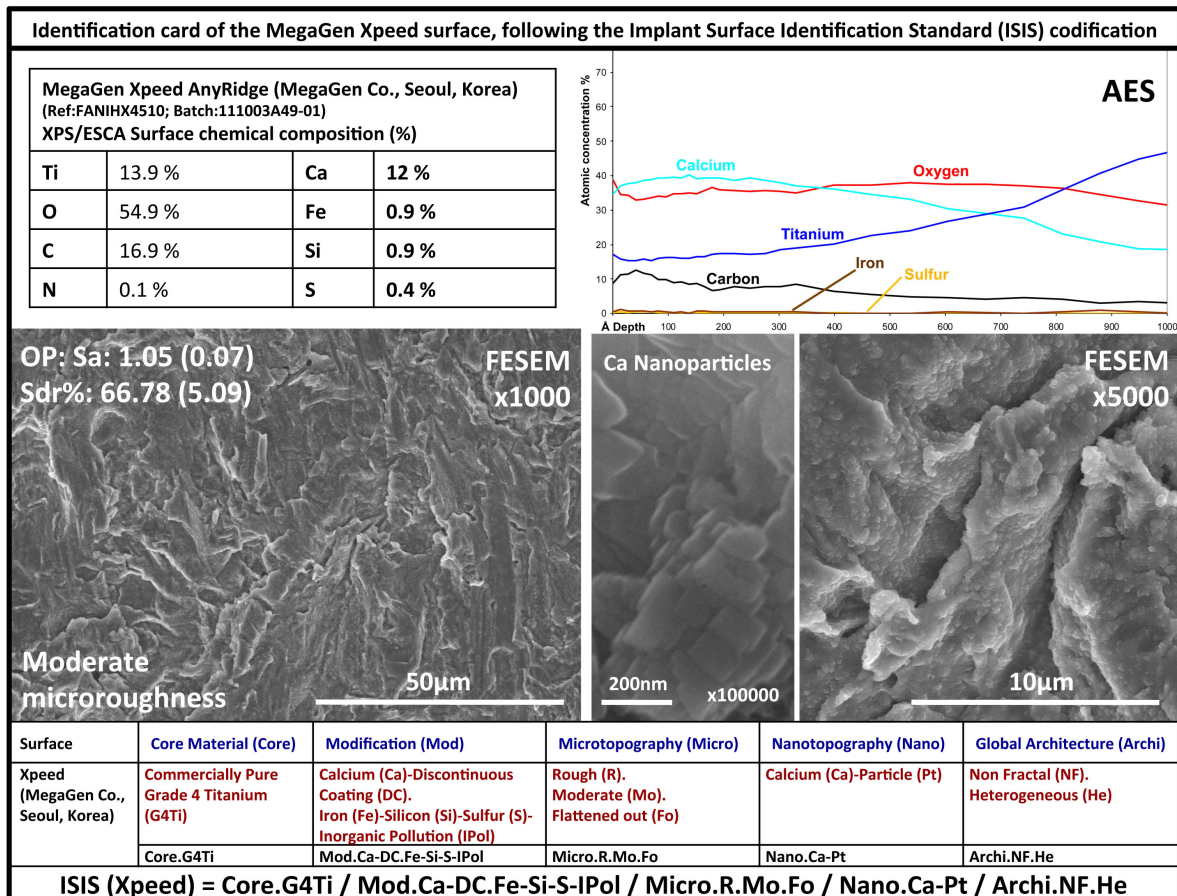


Figure 3. Identification Card of the SLActive surface.





**Figure 4.** Identification Card of the Roxolid SLActive surface.



**Figure 5.** Identification Card of the Xpeed surface.

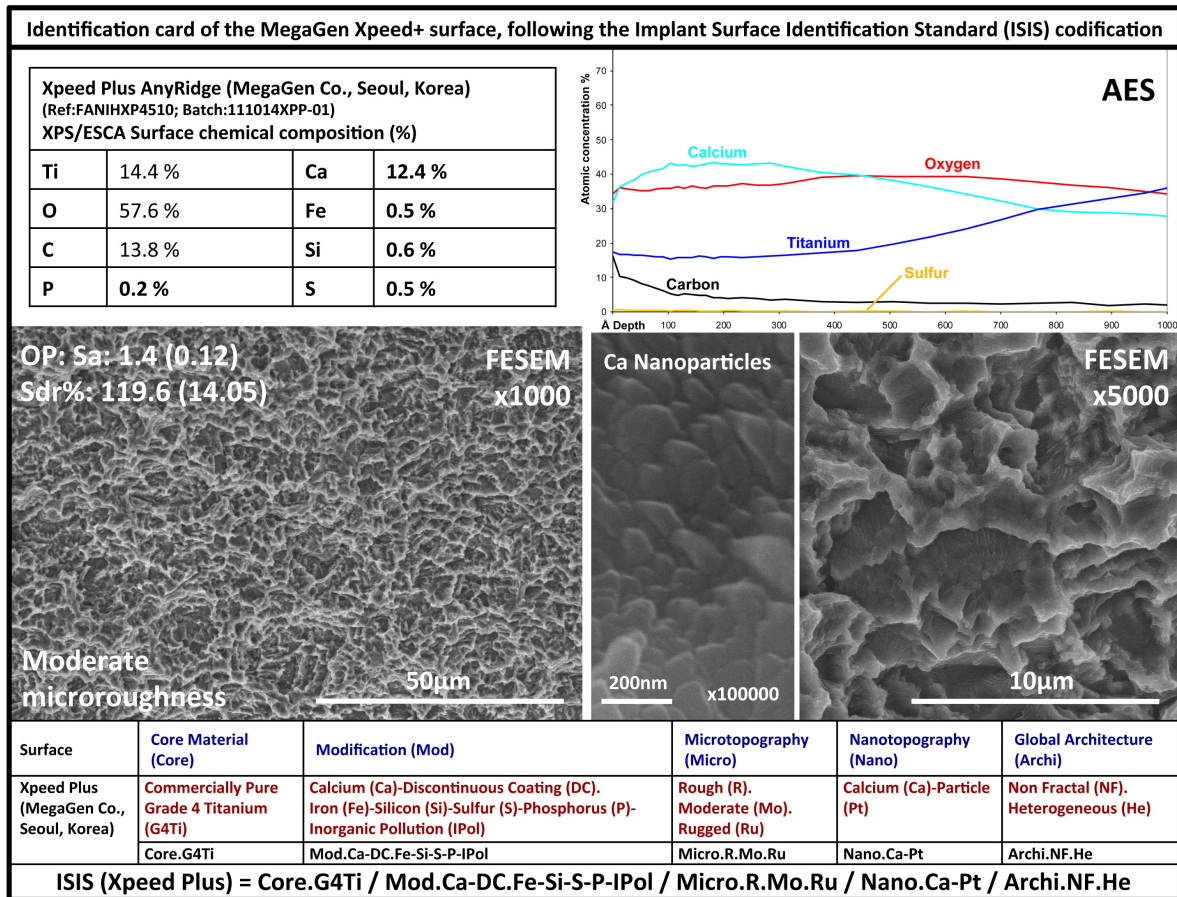


Figure 6. Identification Card of the Xpeed Plus surface.

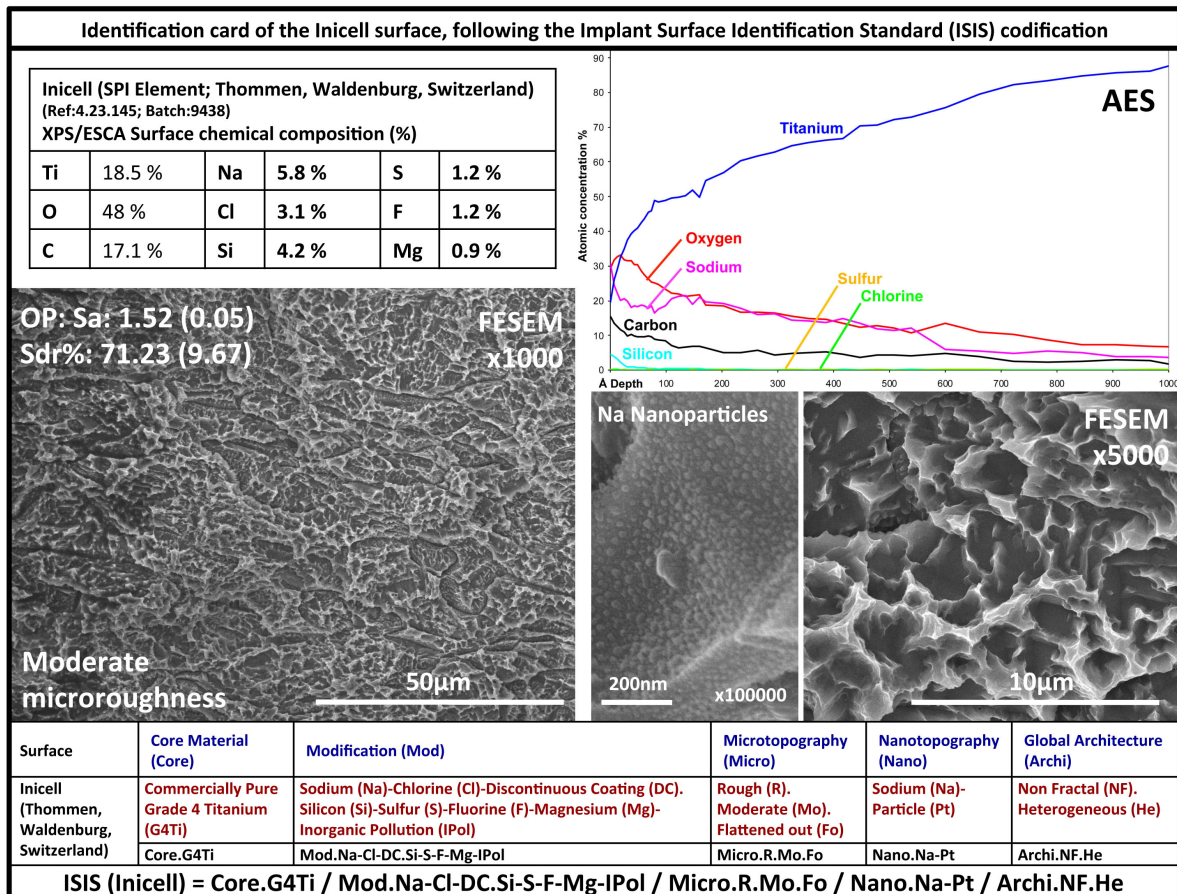
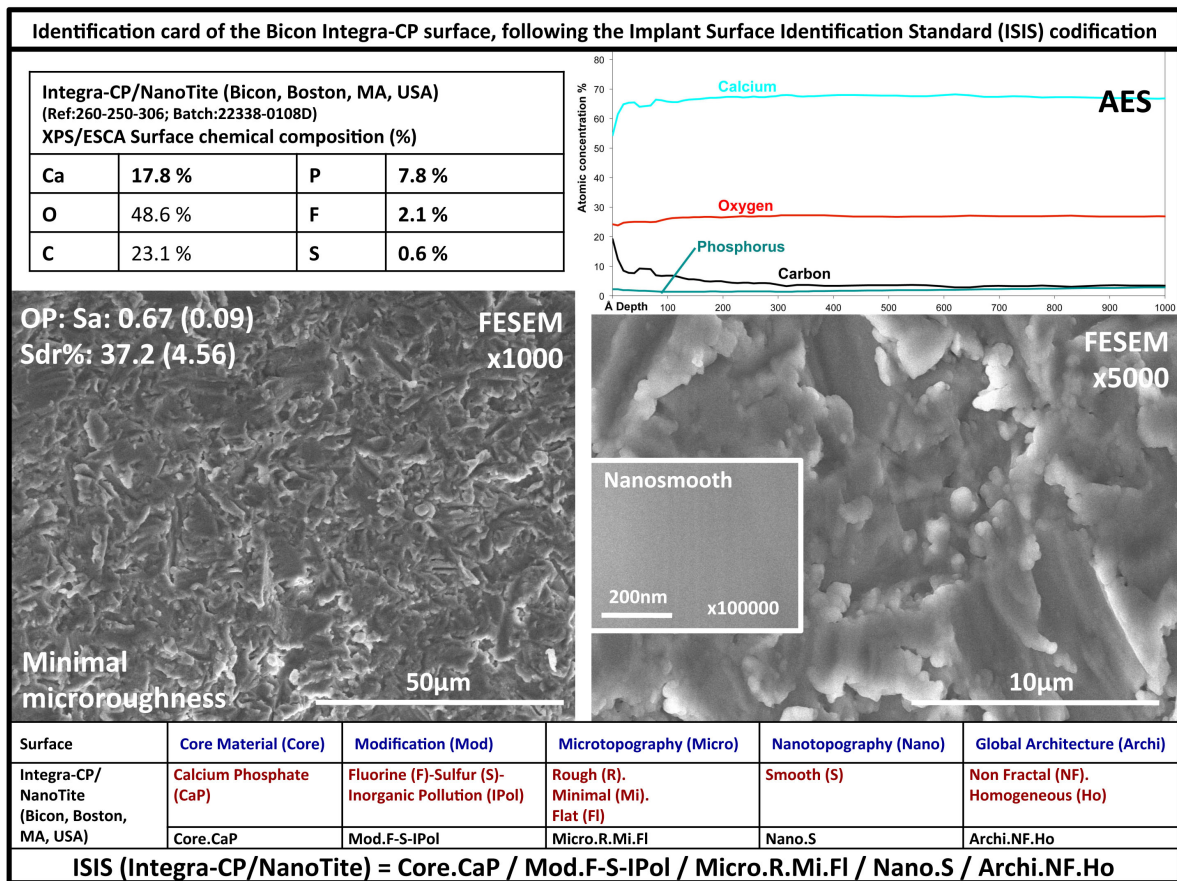
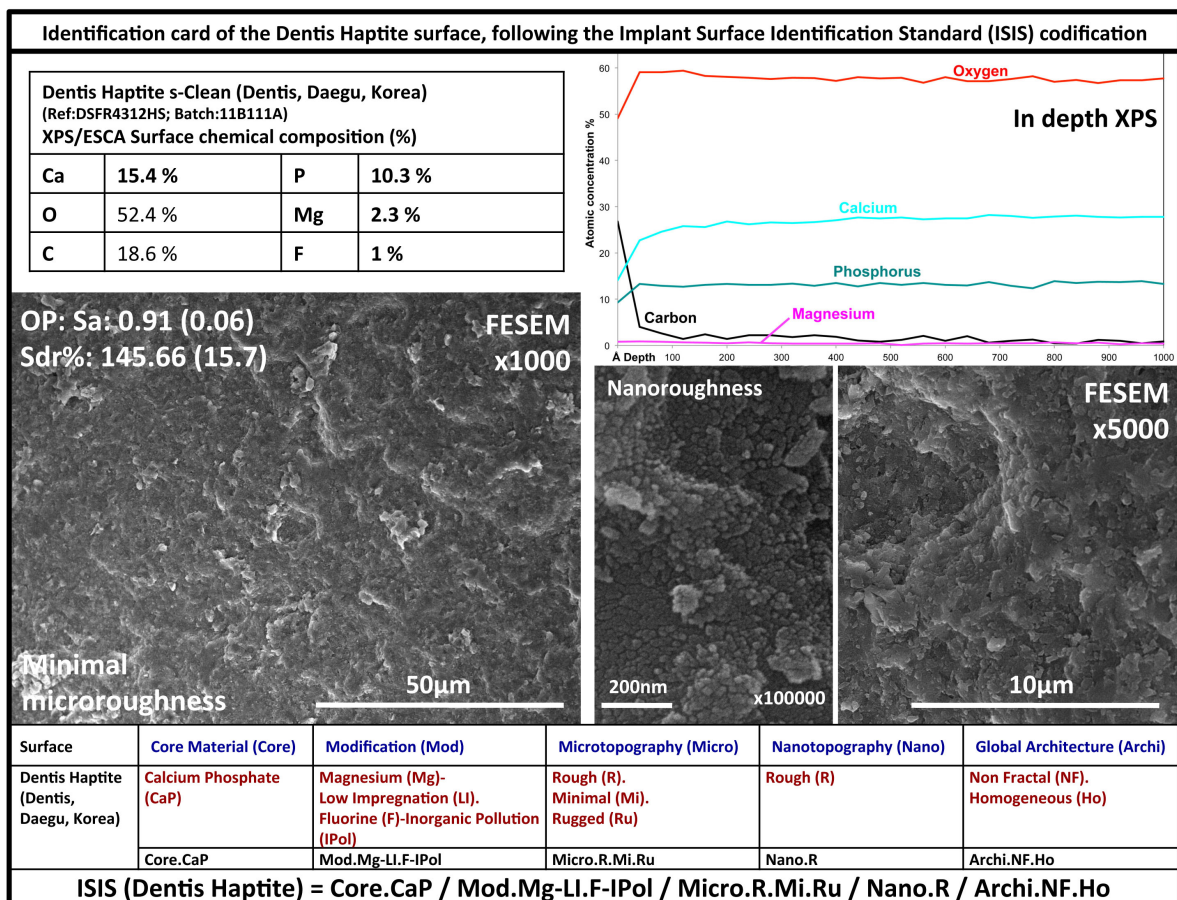


Figure 7. Identification Card of the Inicell surface.





**Figure 8.** Identification Card of the Bicon Integra-CP surface.



**Figure 9.** Identification Card of the Dentis Haptite surface.



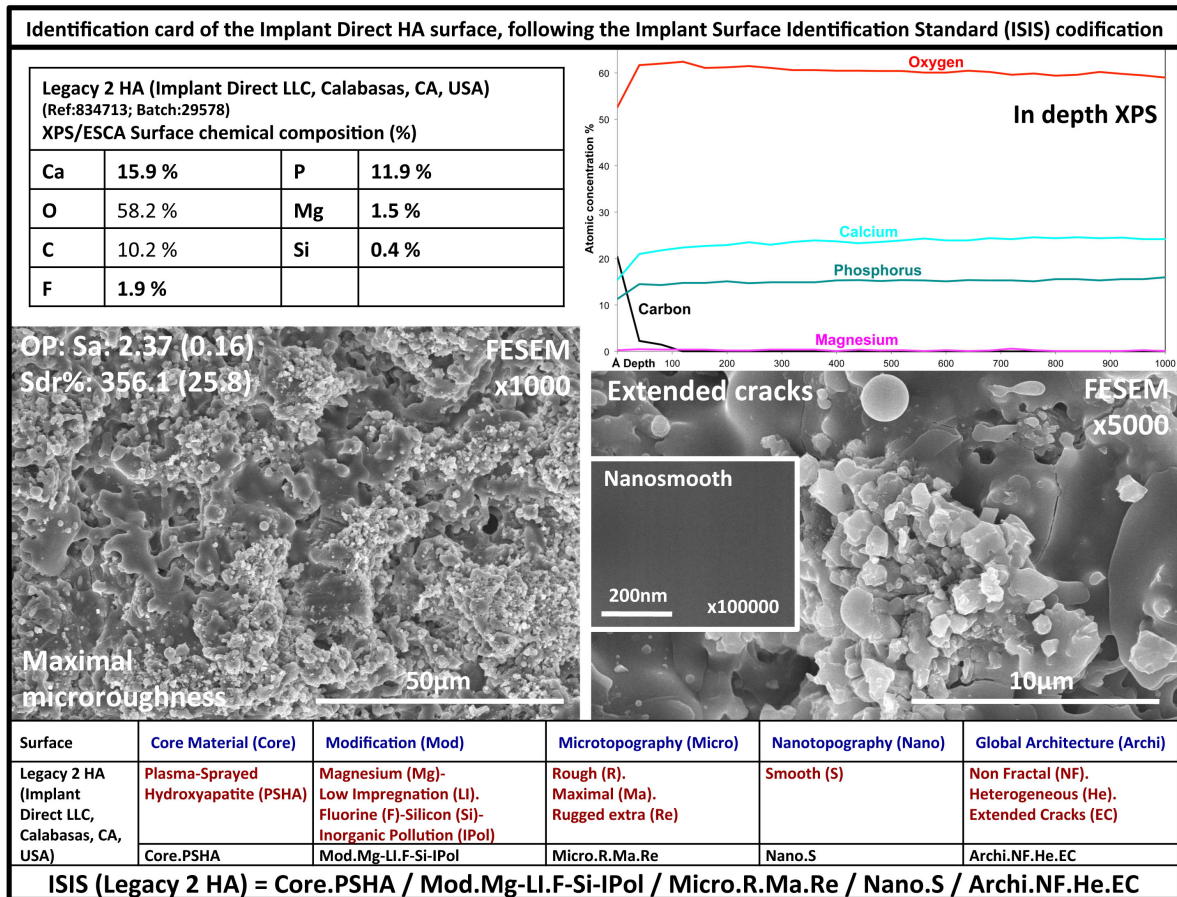


Figure 10. Identification Card of the Legacy 2 HA surface.

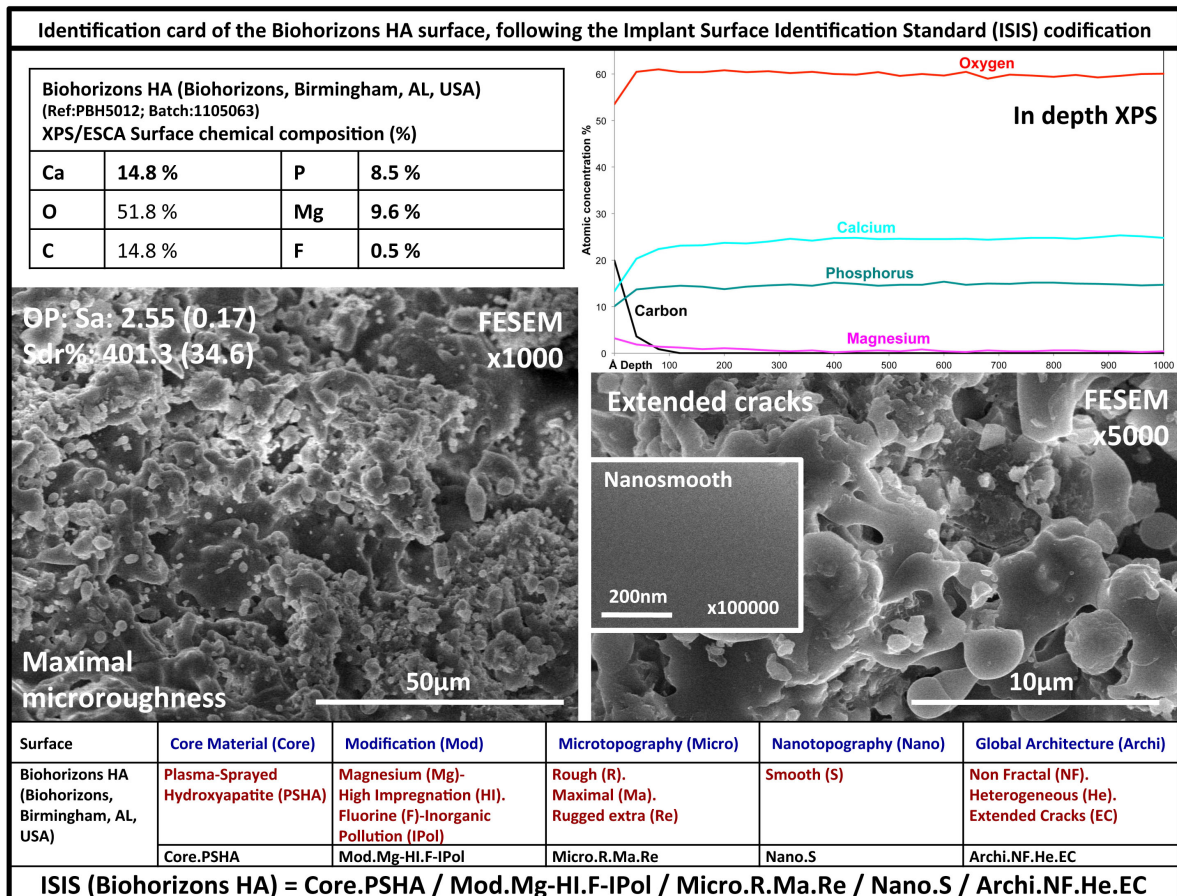
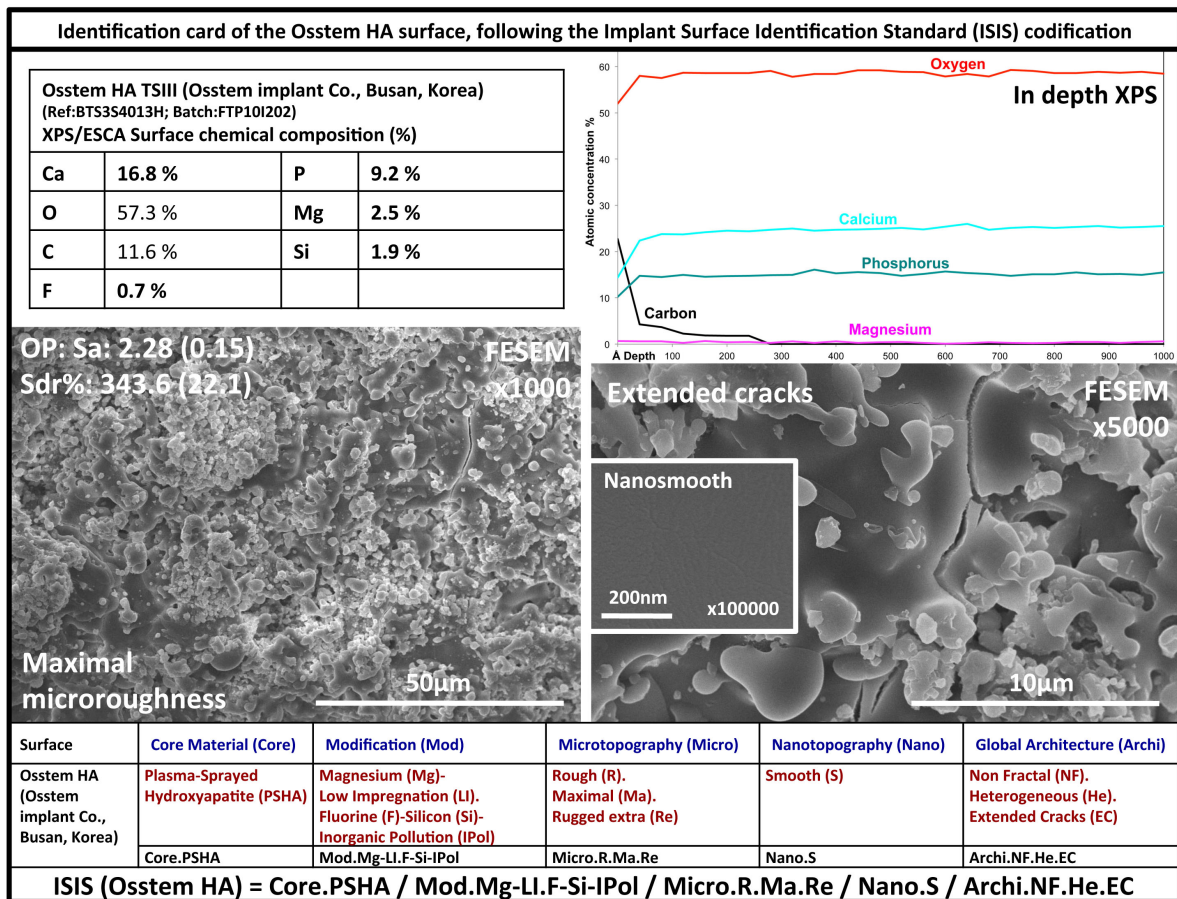
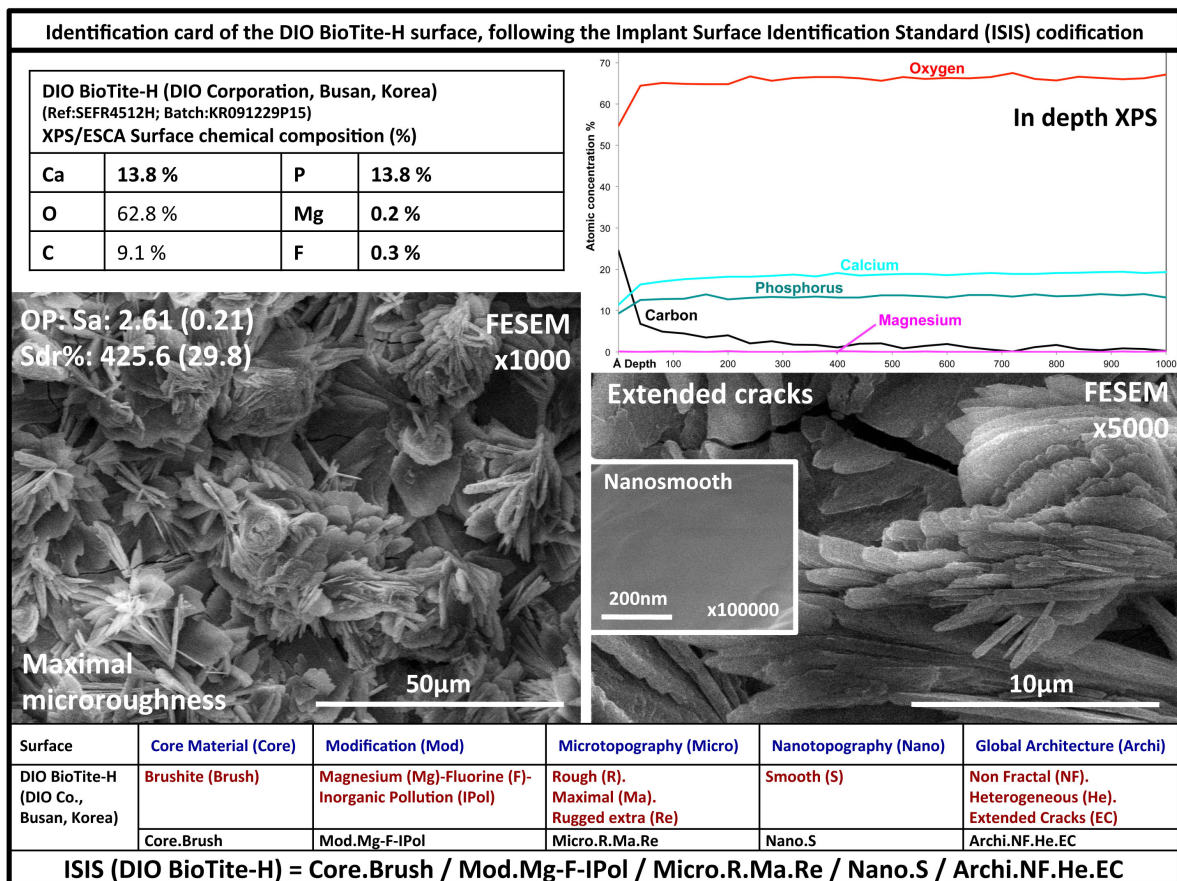


Figure 11. Identification Card of the Biohorizons HA surface.



**Figure 12.** Identification Card of the Osstem HA surface.



**Figure 13.** Identification Card of the DIO BioTite-H surface.



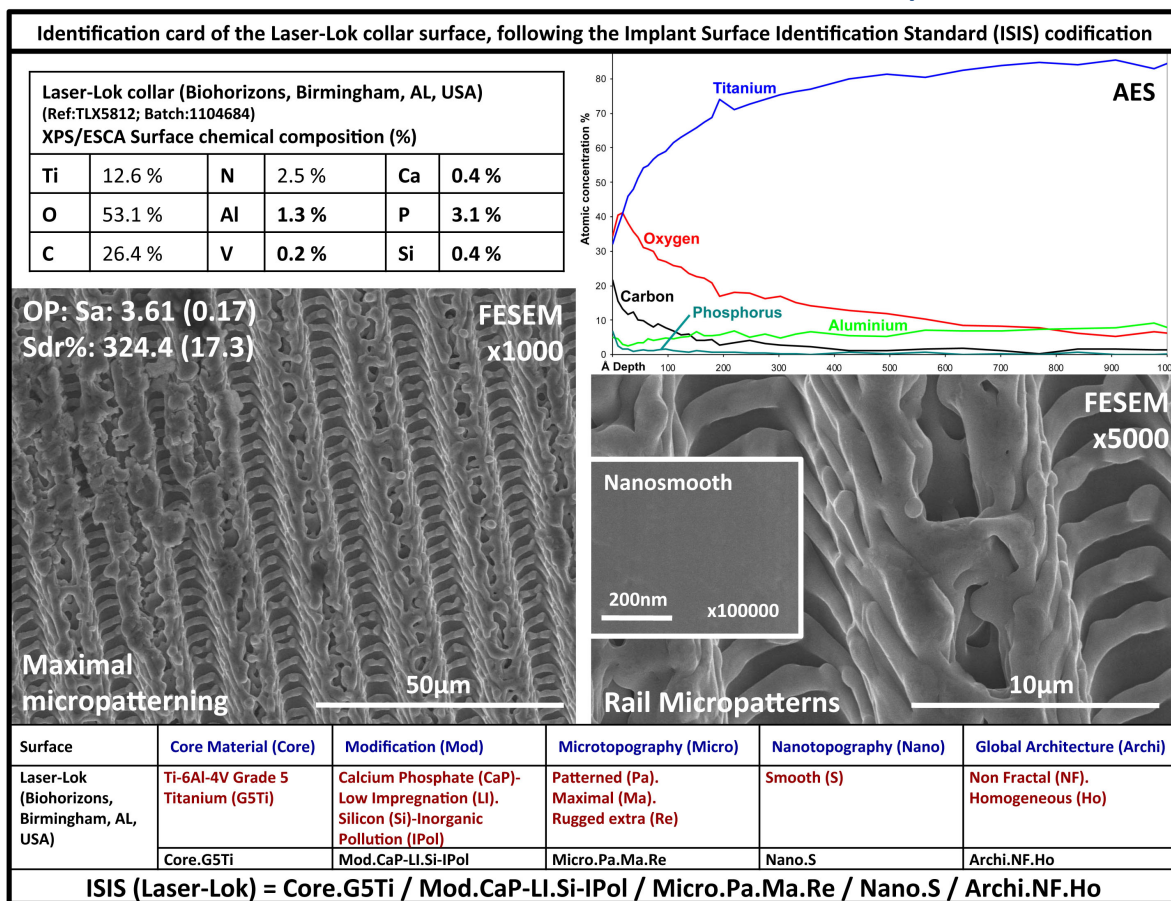


Figure 14. Identification Card of the Laser-Lok collar surface.

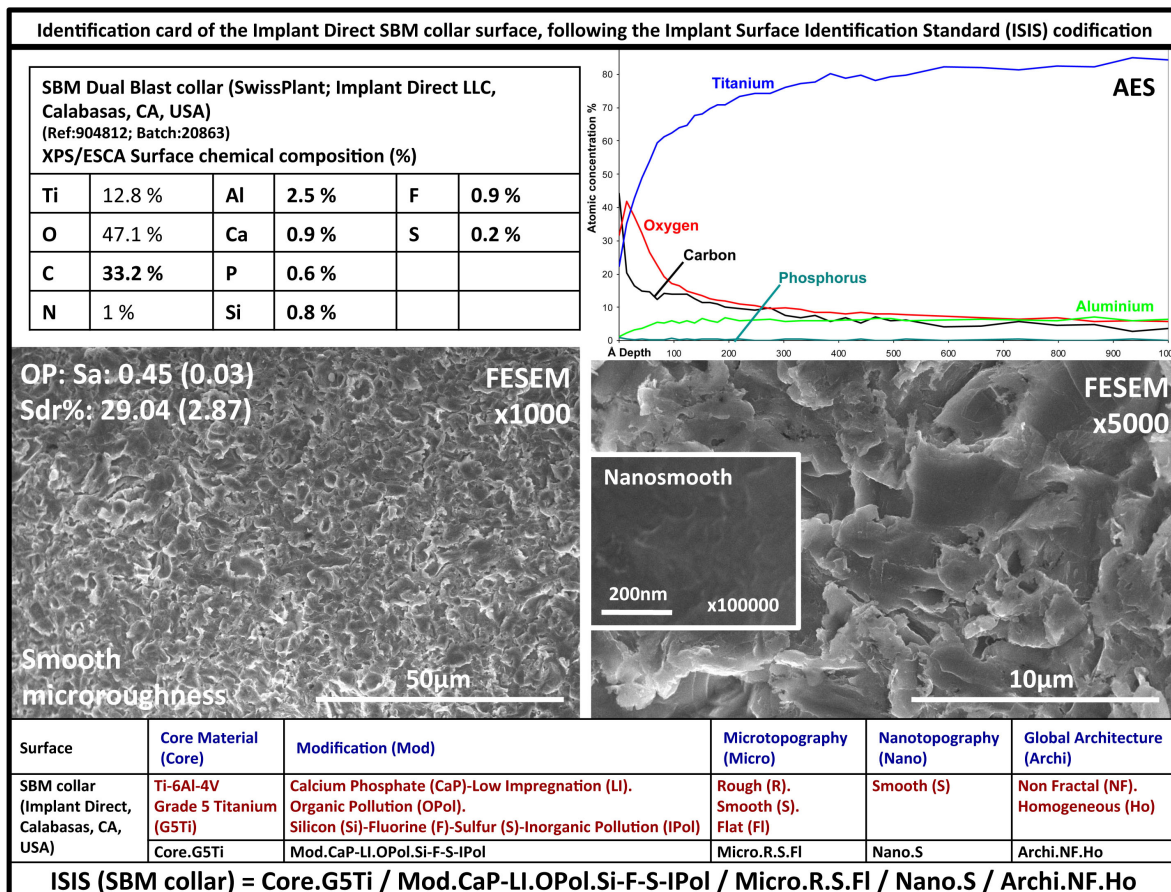
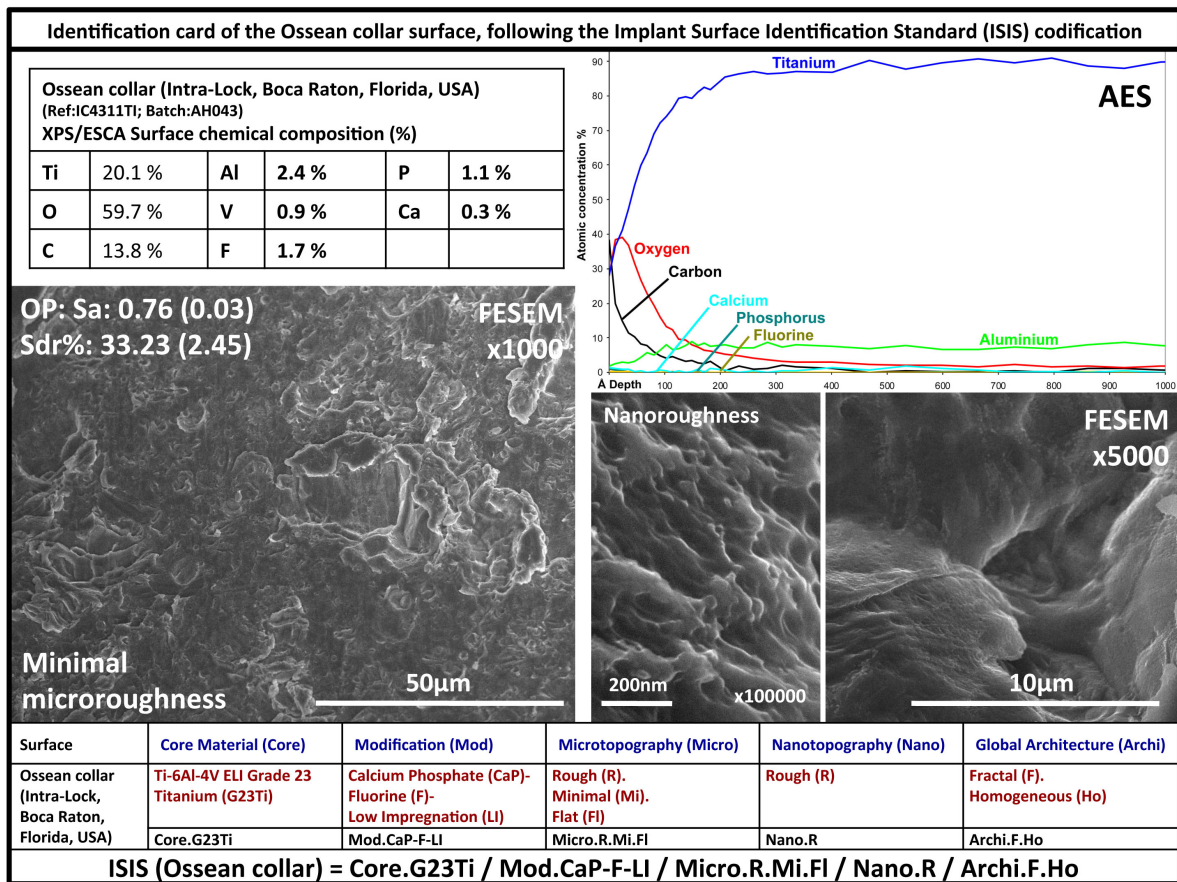
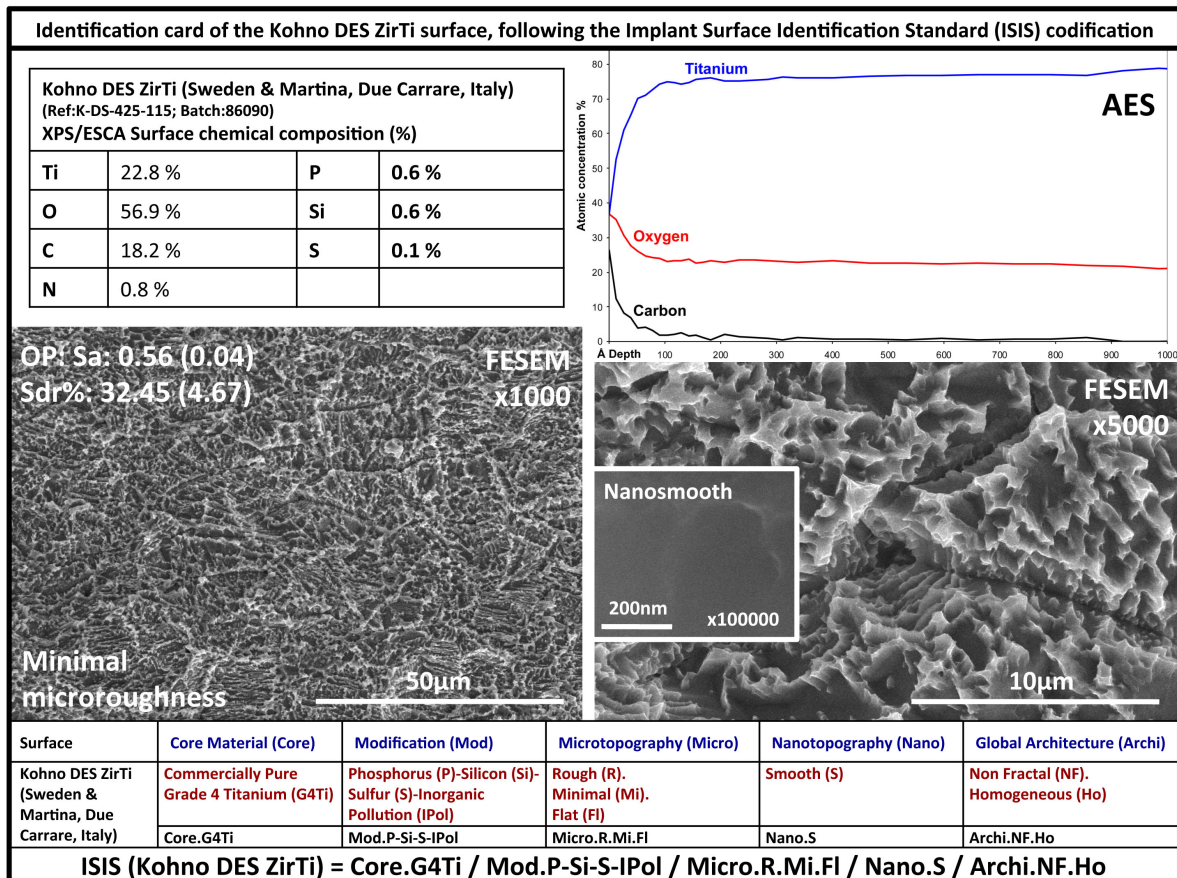


Figure 15. Identification Card of the Implant Direct SBM collar surface.



**Figure 16.** Identification Card of the Ossean collar surface.



**Figure 17.** Identification Card of the Kohno DES ZirTi collar surface.



### 3.4. Collar surfaces (Group 4)

The four samples of this group had only in common to be collar surfaces specifically designed for the interface with the peri-implant cervical tissues (Group 4). Each surface however was following a different production process and even a different concept of biological interaction. Each surface belonged to the technology of a different group: metallurgy modification, RBM-blasting, SIMN, SLA-type. Three of them presented some calcium phosphate impregnation, and 3 presented various forms of pollutions (inorganic or even organic). Surfaces of this group presented different designs of homogeneous microtopography and were in general nanosmooth. Only one surface was using a nanorough and fractal architecture.

Laser-Lok collar (Biohorizons, Birmingham, AL, USA; **Figure 14**) was a surface produced through laser carving of parallel peripheric microchannels all over the implant collar on a grade 5 titanium core. This process was in fact melting the superficial titanium layer to draw the microchannels within the core material. This implant presented in fact 2 very different surfaces: RBT (Resorbable Blast Texture, a RBM-blasted/washed surface) for the implant body (surface of the group 2B, analyzed in the fourth part of this series of articles), and the Laser-Lok surface on the cervical area. The surface was impregnated with low levels of calcium phosphate (CaP), not visible with FE-SEM but homogeneous all over the surface (probably as residues of the RBT processing of the implant body). Some inorganic pollution with silicon was also detected. The very specific microtopography presented deeply melted peripheric rail micropatterns all around the implant collar, quantified as maximal and rugged extra micropatterning. These micropatterns were also nanosmooth, and homogeneous all over the implant collar. This collar surface belonged to the technologies of the group 1, metallurgy modification.

SBM Dual Blast collar (Soluble Blast Media; Implant Direct LLC, Calabasas, CA, USA; **Figure 15**) 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 body was analyzed in the Group 2B (fourth 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 organic pollution (carbon species overcoat) and some inorganic pollution (with silicon, fluorine and sulfur) were also detected. The surface was smooth at the microscale, nanosmooth, and homogeneous all over the implant collar. This collar surface belonged to the technologies of the group 2B, other subtractive process (RBM).

Ossean collar (Intra-Lock, Boca Raton, Florida, USA; **Figure 16**) 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. Ossean collar was a variation of the standard Ossean (used for the implant body and analyzed in the Group 2B, in the fourth part of this series of articles) and was applied on the cervical area of some long collar implants. The surface was impregnated with low levels of calcium phosphate (CaP) and fluorine, not visible with FE-SEM but homogeneous all over the surface. No pollution was detected. The microroughness was minimal and flat, 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. Apart from the difference of core material (as this company used grade 4 or grade 23 titanium depending of the implant design), the main differences between Ossean collar and other forms of Ossean were a flatter microtopography and an additional fluorine low impregnation. This collar



surface belonged to the technologies of the group 2B, other subtractive process (RBM-Subtractive Impregnation Micro/Nanotexturization (SIMN)).

Kohno DES ZirTi (Sweden & Martina, Due Carrare, Italy; **Figure 17**) was a zirconium-blasted/acid-etched titanium surface. The particularity of this implant DES (Dual Engineered Surface) was the presence of 2 very different surfaces: HRPS (High Roughness Plasma Spray) for the 2/3 of the implant from the apex (surface of the group 1, analyzed in the second part of this series of articles), and the ZirTi on the cervical area. Several inorganic pollutions with phosphorus, silicon and sulfur were detected. The surface was minimally microrough, nanosmooth, and homogeneous all over the implant collar. This collar surface belonged to the technologies of the group 2A, main subtractive process (SLA-type).

#### 4. Discussion

The discontinuous nanometric coating of titanium-based surfaces is becoming a frequent evolution since the launch of the Straumann SLActive [7] and 3I NanoTite [8]. The concept is to upgrade a microrough surface with a final chemical coating. The microrough morphology is produced through classical subtractive methods, to promote the classical bone/implant biomechanical interlocking: SLA-type microroughness for Straumann [7], Thommen and Xpeed Plus; RBM-type microroughness for Xpeed [15]; DAE-type microroughness for NanoTite [16]. Then a final layer of nanocrystals is added as a discontinuous coating on the surface, to create a chemical modification and some nanofeatures [17]; the concept of this chemical modification is to promote the bone/implant biochemical interlocking [7,18]. In this sub-group, the chemical coating remained nanometric, less than 100nm thick, and was often discontinuous; therefore the titanium was detectable as core material during the various spectroscopy analyses, was a significant component of the 100nm thickness constituting the surface, and should interact significantly with the bone during the whole osseointegration process. Even if this approach is fashionable and appears as an easy way to upgrade pre-existing microrough surfaces, the clinical significance of this approach is uncertain. The addition of a CaP [8] or Ca [15] nanocrystals coating is debated, as it interferes with the bone catabolism/anabolism cycles, and NanoTite was slowly abandoned due to mixed clinical results [16]. The use of soluble Na-based nanocrystals coating is much easier and many companies are upgrading their SLA-type surfaces through a simple and inexpensive immersion in a NaCl-type physiological solution [7,19], but the rationale behind this concept is also debatable and most literature on the topic was sponsored by companies. It is expected that the fashion of copying SLActive will continue a certain time, and the Group 3A may become as big as the Group 2A (SLA-type) very soon due to this “upgrade”.

One important characteristic of these methods of discontinuous coating was that they produced always very heterogeneous surfaces, where the concentrations of ions or crystals were very different between the various points of the microtopography [4]. With the Ca or CaP coatings, the variations of chemical composition were significant within the peaks and valleys, but there was still a reasonable similarity between areas. With the immersion in NaCl/NaOH or similar solutions, most nanocrystals were randomly deposited on the surface, some area being covered with thick layers, and others without coating, leading to very different chemical modifications and nanofeatures depending on the area; this raises significant concerns about the exact nature of this kind of surfaces and their exact biological effects. It was also important to notice some unexpected inorganic pollution (Fe, Si, S) in all

surfaces of this sub-group, what highlighted the need for some improvements of the industrial processing.

The continuous coating of the core material with a thick micrometric layer of HA or other forms of CaP represents a completely different concept of coating, as in this case the nature of core material is changing. The coating is so thick that the titanium of the implant body remains far from the bone contact, and *de facto* the core material of the surface becomes only the coating itself. The concept of this type of coating is to promote a strong bone/implant biochemical interlocking between the CaP of the surface and the bone mineral phases [9]. PSHA and brushite coatings also create a maximal microroughness due to the deposition technique itself, but this morphology was never advocated to serve as a method of biomechanical interlocking (probably due to the relatively weak mechanical strength of this kind of coatings); these PSHA and brushite coatings always presented very large and significant cracks all over the surface, and this unavoidable characteristic was often pointed out as the source of the abandon of these technologies. In general, we observed some chemical modifications with magnesium in most PSHA surface, as an addition in the PSHA processing. All the implants of this subgroup also presented some inorganic pollution.

In this subgroup, 2 surfaces were a little bit different and required further attention. Bicon Integra-CP was produced through CaP Ion-Beam Assisted Deposition (IBAD) coating [10]. This coating was thicker than 100nm and was therefore micrometric, but it remained much slimmer than the PSHA coating (around 500nm vs 50µm). With this method, the surface was much smoother and without cracks in comparison to PSHA or brushite coatings. Dentis Haptite was also using another interesting method to produce a continuous CaP coating without cracks and with smaller roughness; the method was secret but was described by the company as a form of blasting with HA particles that were impacted and engraved in the titanium core material. The main difference with the Bicon aspect was the final nanorough aspect of the Haptite CaP coating. Haptite was the only surface of this subgroup to present significant nanofeatures, all the others being structurally nanosmooth, due to the production process itself. The Bicon surface associated with specific plateau-designed root implant gave good results [10], but the effect of this kind of surfaces in standard screw implants is debatable. This style of surfaces remains quite rare in the industry nowadays.

The surfaces of the Group 4 were all very different from each other, both in shape, production technology and supporting theoretical concept. The Laser-Lok collar was designed with peripheric micrometric rail patterns [12] produced through laser melting of the titanium surface; this was therefore a surface of the Group 1, where the titanium core material metallurgy was altered to create the surface morphology. This surface did not present chemical modification or nanoroughness, and its supporting concept was only the sealing of the cervical area though this specific peripheric micro-designed attachment ring, where bone and gingival fibers could attach and create a stable frontier area. This concept can however be debated, as a rougher morphology in the implant cervical part may be associated with a higher risk of bacterial contamination [11].

This debate about the choice of the cervical roughness is very important, as many companies decided to reduce the roughness at the cervical part to reduce the risk of bacterial contamination, peri-implantitis and bone resorption [11]. For example, the ZirTi surface was created as a complement cervical surface for the traditional HRPS (High Roughness Plasma Sprayed) of Sweden-Martina. HRPS was a Titanium Plasma-Sprayed TPS surface and was therefore considered too rough for the cervical part, so this company created the concept of DES (Dual Engineered Surface) implant, where most of the implant from the apical was

covered with HRPS for higher bone biomechanical anchorage and the cervical part was covered with a much smoother surface ZirTi. ZirTi was a zirconium-blasted acid-etched surface (Group 2A), minimally rough and presenting typical patterns from a flattened out SLA-type surface, in order to promote a significant peri-implant cervical biomechanical sealing [13]. This surface did not present chemical modification or nanoroughness to promote this sealing, the whole concept was based on a strict biomechanical anchorage.

A similar strategy of reduction of the implant microroughness for the cervical parts of the implants was also found with the Implant Direct and Intra-Lock samples, each company using however a slightly different concept at the chemical and nanoscale. Both were RBM-type surfaces (Group 2B), Ossean being also a Subtractive Impregnated Micro/Nanotextured (SIMN) surface [20].

Implant Direct proposed a similar approach of 2-surface combination with the SBM (Soluble Blast Media) Dual Blast surface, where the implant body was prepared through a classical RBM-type processing (analyzed in the part 4 of this series of articles, Group 2B) and the implant collar was treated with the same RBM but in softer conditions to reach a much smoother morphology at the microscale. The surface remained nanosmooth, but the use of a RBM processing allowed to impregnate the surface with some CaP, this chemical modification being expected to stimulate the healing of peri-implant tissues, even if no clear experimental validation or feedback of experience could be found in the literature.

Intra-Lock also designed a specific version of the Ossean surface for the collar area of its implant, following the same process producing a CaP low impregnation and a homogeneous nanoroughness [14]. The main difference with the classical Ossean surface (used on the implant body)[20] was characterized as a more flattened out morphology at the microscale and the addition of some fluorine impregnation to stimulate the cell and matrix growth and proliferation. This collar surface was therefore also a Subtractive Impregnated Micro/Nanotextured (SIMN, Group 2B) surface. The concept of this approach was to combine: a minimal microroughness for the biomechanical anchorage and the reduction of the bacterial contamination risk; a stimulation of bone and soft tissue healing at the collar interface through the combination of nanoroughness and chemical impregnation. Due to this homogeneous 3-level one-dimension texturization (chemical, nano, micro), this surface was the only one to appear fractal, following the previous definition [5]. Some good experimental results were described in the literature with this surface combination [14], and the SIMN technology represents an interesting biologically-driven evolution of the collar surface designs.

Finally, some inorganic and organic pollutions were found on 3 of these 4 samples (only the Ossean surface did not present any pollution), highlighting the needs for some industrial improvements in the future. As a conclusion, the collar surfaces are based on the classical 3 groups of surfaces (modification of titanium metallurgy, subtraction through blasting and etching, coating), and they represent an interesting and significant path of research in the field in the coming years.

## 5. Conclusion

The coating of titanium surfaces with nanometric chemical layers is a quite new and fashionable approach, even if some methods have already been abandoned with CaP discontinuous coating. The method with Na-based solutions (initiated by SLActive) appears as an easy way to add some chemical and nano-features on the classical microrough surfaces, to combine their biomechanical interlocking with some chemically-driven biochemical

interlocking. The main concern of this approach is the strong heterogeneous aspect of these surfaces and the absence of independent validation of the relevance of such soluble temporary Na coating; the concept is however spreading extensively nowadays. On the other hand, thick coatings with CaP or HA are quite rare nowadays, due to the reported risks of coating fractures and delaminations of the cracked surfaces. Only few companies are still promoting this concept with some specific implant designs. Finally, the implant collars are the new Frontier of surface evolutions, and the use of specialized collar surfaces is still rare but developing quickly; however, in the absence of clear consensus, each company is promoting its own concept, through the adaptation of the various surface technologies previously developed: strong micropatterning or microsmooth surfaces, with or without chemical modification and/or nanotexturization. In this domain, the development of SIMN surfaces seems again the most interesting path of research, and it is expected that the surfaces of the Group 4 will become a key topic of investigation in the future. Moreover, the frequent presence of inorganic or organic contaminants on the many tested products revealed that some improvements are still needed to increase the industrial quality.

### 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

DMDE, MDC, BSK, JPB and GS were leading the general organization, surface analyses and main financial support of this considerable international project. All authors participated to the development of a consensual analytical process, to the collection of samples and data, and to the elaboration of the manuscript.

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