

Research article

Identification card and codification of the chemical and morphological characteristics of 62 dental implant surfaces. Part 3: sand-blasted/acid-etched (SLA type) and related surfaces (Group 2A, main 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 third part, surfaces produced through the main subtractive process (sand-blasting/acid-etching, SLA-type and related) were investigated.

Materials and Methods. Eighteen different implant surfaces were characterized: Straumann SLA (ITI Straumann, Basel, Switzerland), Ankylos (Dentsply Friadent, Mannheim, Germany), Xive S (Dentsply Friadent, Mannheim, Germany), Frialit (Dentsply Friadent, Mannheim, Germany), Promote (Camlog, Basel, Switzerland), Dentium Superline (Dentium Co., Seoul, Korea), Osstem SA (Osstem implant Co., Busan, Korea), Genesis (GC Corporation, Tokyo, Japan), Aadvia (GC Corporation, Tokyo, Japan), MIS Seven (MIS Implants Technologies, Bar Lev, Israel), ActivFluor (Blue Sky Bio, Grayslake, IL, USA), Tekka SA2 (Tekka, Brignais, France), Twinkon Ref (Tekka, Brignais, France), Bredent OCS blueSKY (Bredent Medical, Senden, Germany), Magitech MS2010 (Magitech M2I, Levallois-Perret, France), EVL Plus (SERF, Decines, France), Alpha Bio (Alpha Bio Tec Ltd, Petach Tikva,

Israel), Neoporos (Neodent, Curitiba, Brazil). 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 18 different surfaces of this group, 11 were based on a commercially pure titanium (grade 2 or 4) and 7 on a titanium-aluminium alloy (grade 5 or grade 23 ELI titanium). 4 surfaces presented some chemical impregnation of the titanium core, and 5 surfaces were covered with residual alumina blasting particles. 15 surfaces presented different degrees of inorganic pollutions, and 2 presented a severe organic pollution overcoat. Only 3 surfaces presented no pollution (and also no chemical modification at all): GC Aadva, Genesis, MIS Seven. From a morphological standpoint, all surfaces were microrough, with different microtopographical aspects and values. All surfaces were nanosmooth, and therefore presented no significant and repetitive nanostructures. 14 surfaces were homogeneous and 4 heterogeneous. None of them 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. The SLA-type surfaces have specific morphological characteristics (microrough, nanosmooth, with rare and in general accidental chemical modification) and are the most frequent surfaces used in the industry. However they present different designs, and pollutions are often detected (with blasting/etching residues particularly). Users should be aware of these specificities if they decide to use these products.

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 [2,3]. 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 [4]. 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 [5]. This card describes the surface chemical composition and morphological characteristics of each surface [6,7]. This standardized codification system allows to clarify the identity of each surface and to easily sort their differences [5]. 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, which can be associated with some chemical modifications. All the surfaces of this category are presenting various levels of microroughness [6]. Many versions of this approach exist, but the most common nowadays is the combination of sand-blasting and acid-etching [8]. The titanium core material is blasted with hard microparticles of alumina (Al_2O_3) to carve a microtopography on the surface, and then etched with strong acid solutions to clean the blasting residues and finish the design of the microgeometry. The many versions of this process are regrouped in the Group 2A, and the most frequent method nowadays was termed SLA, i.e. Sand-blasted Large-grit Acid-etched (commercial name from Straumann, Basel, Switzerland)[9]. This method was frequently copied on the market, and many variations exist leading to different morphological designs on the microscale [6].

In this third part, the chemical and morphological characteristics of 18 implant surfaces (available on the market) from the group 2A were investigated and described through a simple and clear identification (ID) card for each surface, following the ISIS system terminology and classification. The group 2A gathered all surfaces produced through a subtractive processing to carve the surface morphology on the core material, coupling sand-blasting and acid-etching (SLA type).

2. Materials and Methods

2.1. Samples

Eighteen different implant surfaces of the Group 2A have been investigated: Straumann SLA (ITI Straumann, Basel, Switzerland), Ankylos (Dentsply Friadent, Mannheim, Germany), Xive S (Dentsply Friadent, Mannheim, Germany), Frialit (Dentsply Friadent, Mannheim, Germany), Promote (Camlog, Basel, Switzerland), Dentium Superline (Dentium Co., Seoul, Korea), Osstem SA (Osstem implant Co., Busan, Korea), Genesisio (GC Corporation, Tokyo, Japan), Aadvia (GC Corporation, Tokyo, Japan), MIS Seven (MIS Implants Technologies, Bar Lev, Israel), ActivFluor (Blue Sky Bio, Grayslake, IL, USA), Tekka SA2 (Tekka, Brignais, France), Twinkon Ref (Tekka, Brignais, France), Bredent OCS blueSKY (Bredent Medical, Senden, Germany), Magitech MS2010 (Magitech M2I, Levallois-Perret, France), EVL Plus (SERF, Decines, France), Alpha Bio (Alpha Bio Tec Ltd, Petach Tikva, Israel), Neoporos (Neodent, Curitiba, Brazil). 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 GC 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 $\text{AlK}\alpha$ 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 TiO₂: 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)[6]. 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 [6], 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 18 different surfaces of this group, 11 were based on a commercially pure titanium (grade 2 or 4) and 7 on a titanium-aluminium alloy (grade 5 or grade 23 ELI titanium). 4 surfaces presented some chemical impregnation of the titanium core, and 5 surfaces were covered with residual alumina blasting particles. 15 surfaces presented different degrees of inorganic pollutions, and 2 presented a severe organic pollution overcoat. Only 3 surfaces presented no pollution (and also no chemical modification at all).

From a morphological standpoint, all surfaces were microrough, with different microtopographical aspects and values. All surfaces were nanosmooth, and therefore presented no significant and repetitive nanostructures. 14 surfaces were homogeneous and 4 heterogeneous. None of them 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. Descriptions of the surfaces

Straumann SLA (Sand-blasted, Large-grit, Acid-etched; ITI Straumann, Basel, Switzerland; **Figure 1**) was a sand-blasted/acid-etched surface. Some inorganic pollution with silicon was detected. The surface was moderately microrough, nanosmooth, and homogeneous all over the implant.

Ankylos (Dentsply Friadent, Mannheim, Germany; **Figure 2**) was a sand-blasted/acid-etched surface. The surface was covered with alumina particles (Al_2O_3) and many other inorganic pollutions were detected with sodium, fluorine, calcium, phosphorus (as phosphate), zinc, chloride and sulfur (as sulfate). The surface was moderately microrough and nanosmooth, but heterogeneous all over the implant (particularly because of the many residues). The surface tested here was an early version of Ankylos; the latest version was in theory the same than Xive and Frialit.

Xive S (Dentsply Friadent, Mannheim, Germany; **Figure 3**) was a sand-blasted/acid-etched surface (process called Friadent Plus). Some inorganic pollutions with calcium and sulfur were detected. The surface was maximally microrough, nanosmooth, and homogeneous all over the implant.

Frialit (Dentsply Friadent, Mannheim, Germany; **Figure 4**) was a sand-blasted/acid-etched surface (process called Friadent Plus). Some inorganic pollutions with silicon and fluorine were detected. The surface was moderately microrough, nanosmooth, and

homogeneous all over the implant. Frialit and Xive were supposed in theory to be almost the same surface, while they presented practically some clear differences.

Camlog Promote (Camlog, Basel, Switzerland; **Figure 5**) was a sand-blasted/acid-etched surface. Some inorganic pollutions with zinc and calcium were detected. The surface was moderately microrough, nanosmooth, and homogeneous all over the implant.

Dentium Superline (Dentium Co., Seoul, Korea; **Figure 6**) was a sand-blasted/acid-etched surface. Some inorganic pollution with silicon was detected. The surface was moderately microrough, nanosmooth, and homogeneous all over the implant.

Osstem SA (Osstem implant Co., Busan, Korea; **Figure 7**) was a sand-blasted/acid-etched surface. Some inorganic pollution with silicon was detected. The surface was maximally microrough, nanosmooth, and homogeneous all over the implant.

Genesio (GC Corporation, Tokyo, Japan; **Figure 8**) was a sand-blasted/acid-etched surface. No pollution or chemical modification was detected. The surface was maximally microrough, nanosmooth, and homogeneous all over the implant.

Aadva (GC Corporation, Tokyo, Japan; **Figure 9**) was a sand-blasted/acid-etched surface on a grade 5 titanium core. No pollution or chemical modification was detected. The surface was moderately microrough, nanosmooth, and homogeneous all over the implant.

MIS Seven (MIS Implants Technologies, Bar Lev, Israel; **Figure 10**) was a sand-blasted/acid-etched surface on a grade 23 ELI (Extra Low Interstitials) titanium core. No pollution or chemical modification was detected. The surface was moderately microrough, nanosmooth, and homogeneous all over the implant.

ActivFluor (Blue Sky Bio, Grayslake, IL, USA; **Figure 11**) was a sand-blasted/acid-etched surface on a grade 5 titanium core. Some inorganic pollutions with silicon and phosphorus were detected. The surface was minimally microrough, nanosmooth, and homogeneous all over the implant.

Tekka SA2 (Tekka, Brignais, France; **Figure 12**) was a sand-blasted/acid-etched surface on a grade 5 titanium core. Several inorganic pollutions with calcium, phosphorus, silicon, iron and barium were detected. The surface was moderately microrough, nanosmooth, and homogeneous all over the implant.

Twinkon Ref (Tekka, Brignais, France; **Figure 13**) was a sand-blasted surface on a grade 5 titanium core. The surface appeared impregnated with low levels of calcium, and covered with alumina particles (Al_2O_3) and a thick organic pollution (thick carbon overcoat all over the implant). Some other inorganic pollutions were detected with silicon, sulfur (as sulfate), chloride and zinc. The surface was minimally microrough, nanosmooth, and heterogeneous all over the implant.

Bredent OCS blueSKY (Bredent Medical, Senden, Germany; **Figure 14**) was a sand-blasted/acid-etched surface. A calcium residual impregnation and several inorganic pollutions with magnesium, silicon and barium were detected. The surface was moderately microrough, nanosmooth, and homogeneous all over the implant.

Magitech MS2010 (Magitech M2I, Levallois-Perret, France; **Figure 15**) was a sand-blasted/acid-etched surface on a grade 5 titanium core. It was impregnated with low levels of calcium and covered with small alumina (Al_2O_3) particles. Several inorganic pollutions with silicon, fluorine, zinc, magnesium and sulfur were detected. The surface was minimally microrough, nanosmooth, and heterogeneous all over the implant.

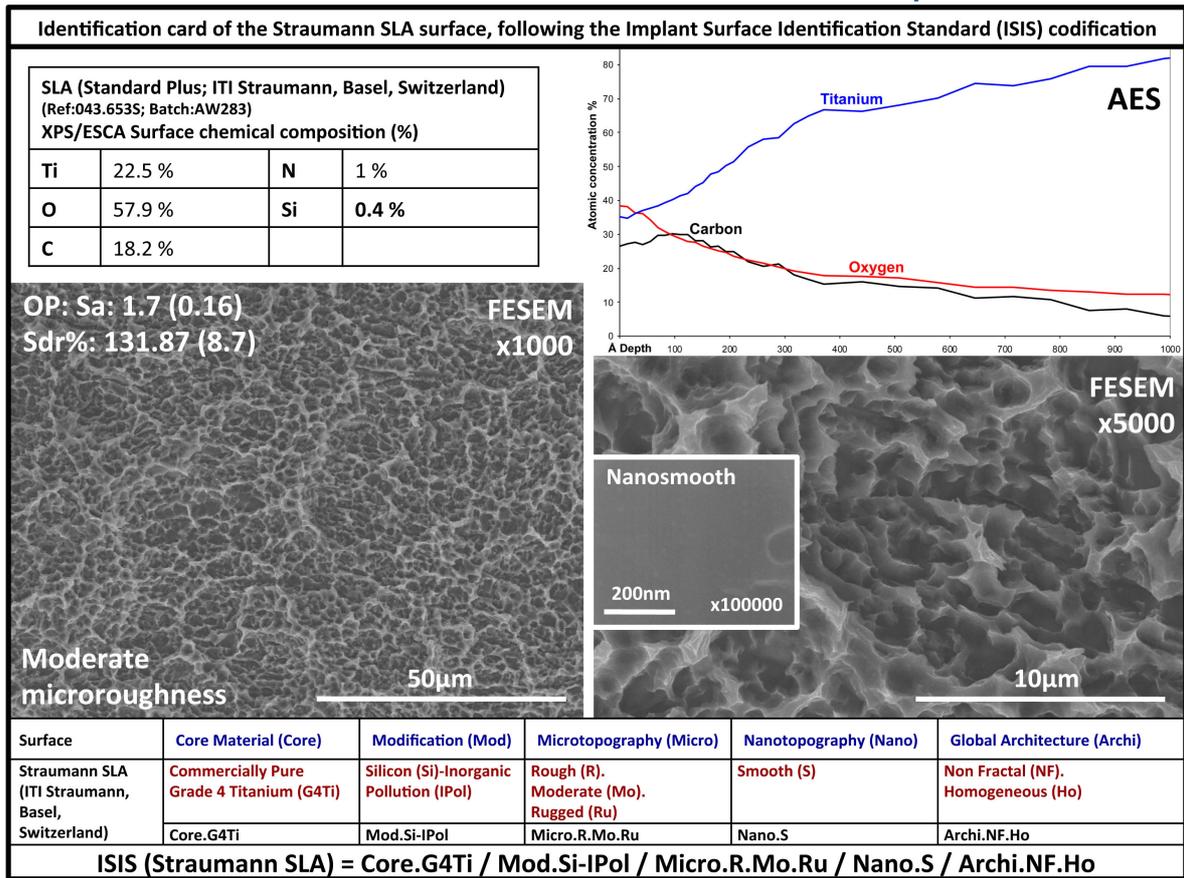


Figure 1. Identification Card of the Straumann SLA surface.

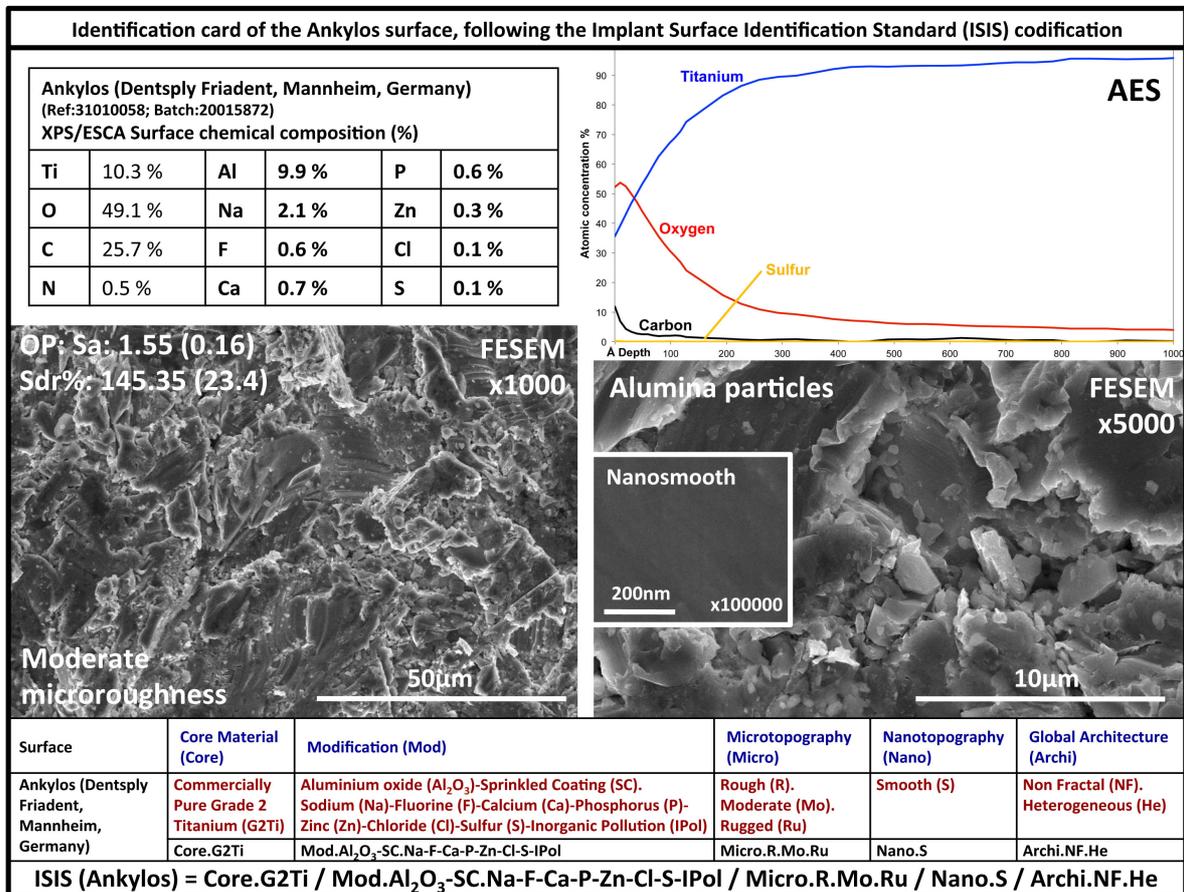


Figure 2. Identification Card of the Ankylos surface.

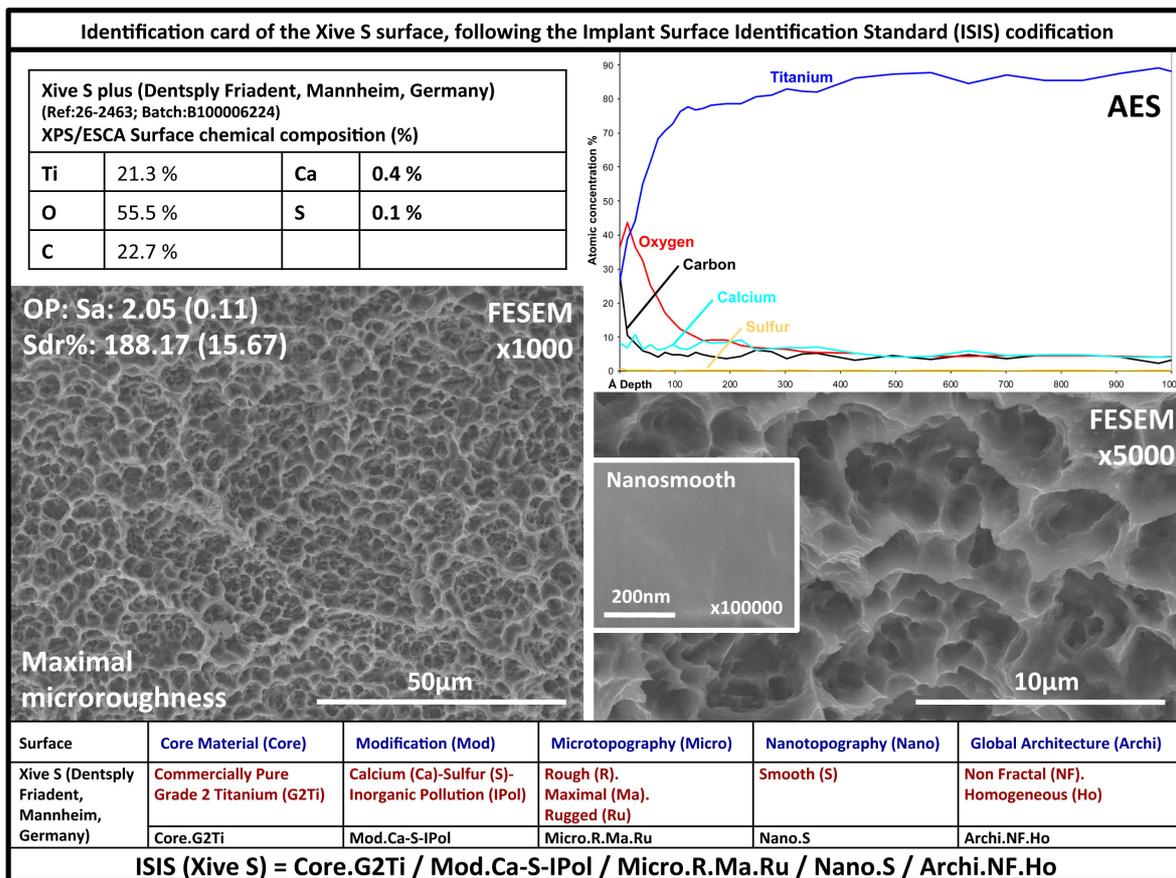


Figure 3. Identification Card of the Xive S surface.

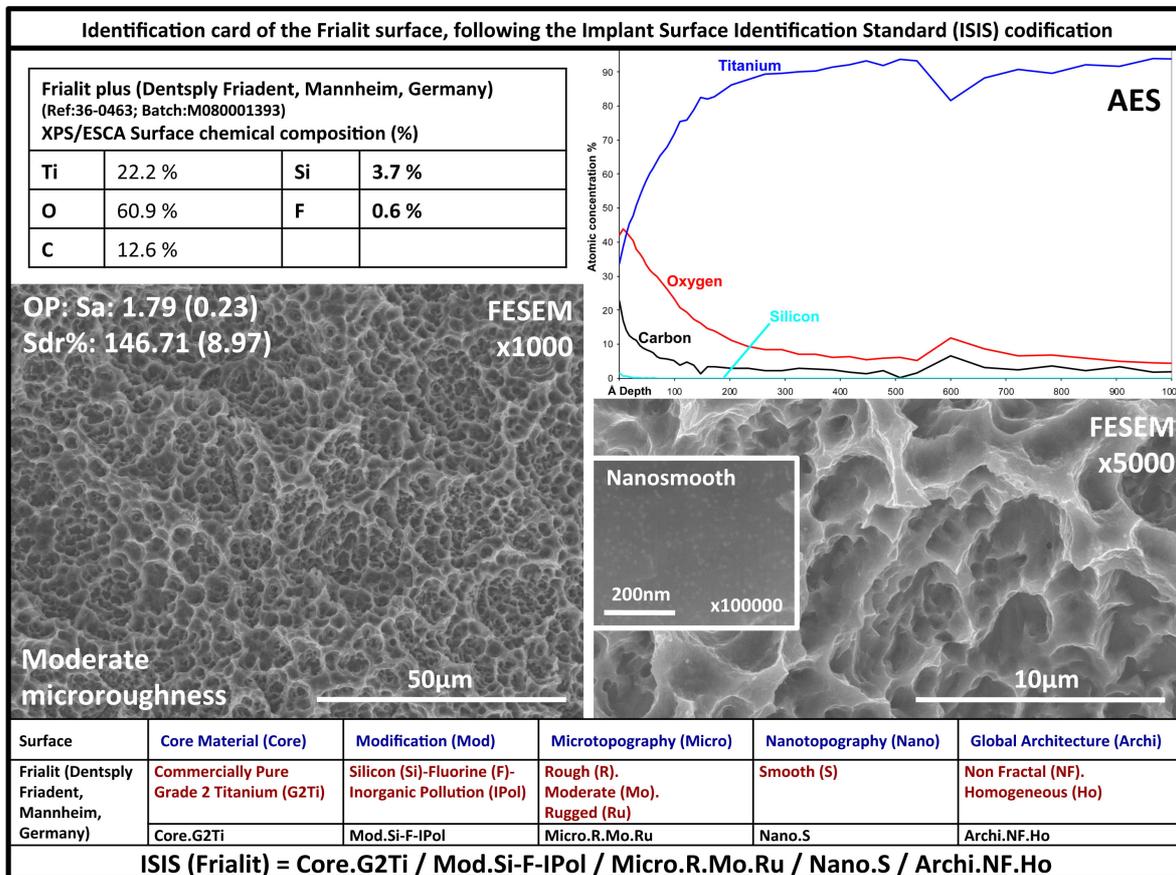


Figure 4. Identification Card of the Frialit surface.

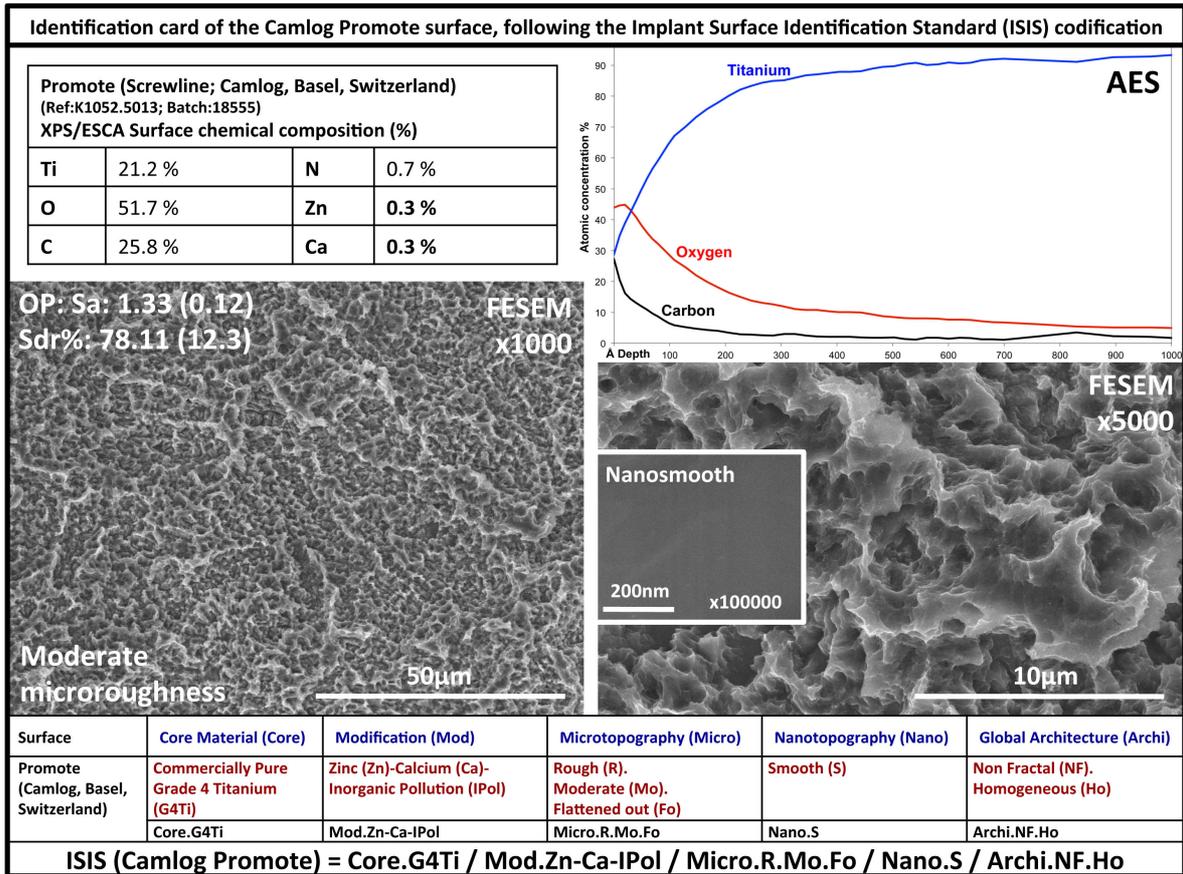


Figure 5. Identification Card of the Camlog Promote surface.

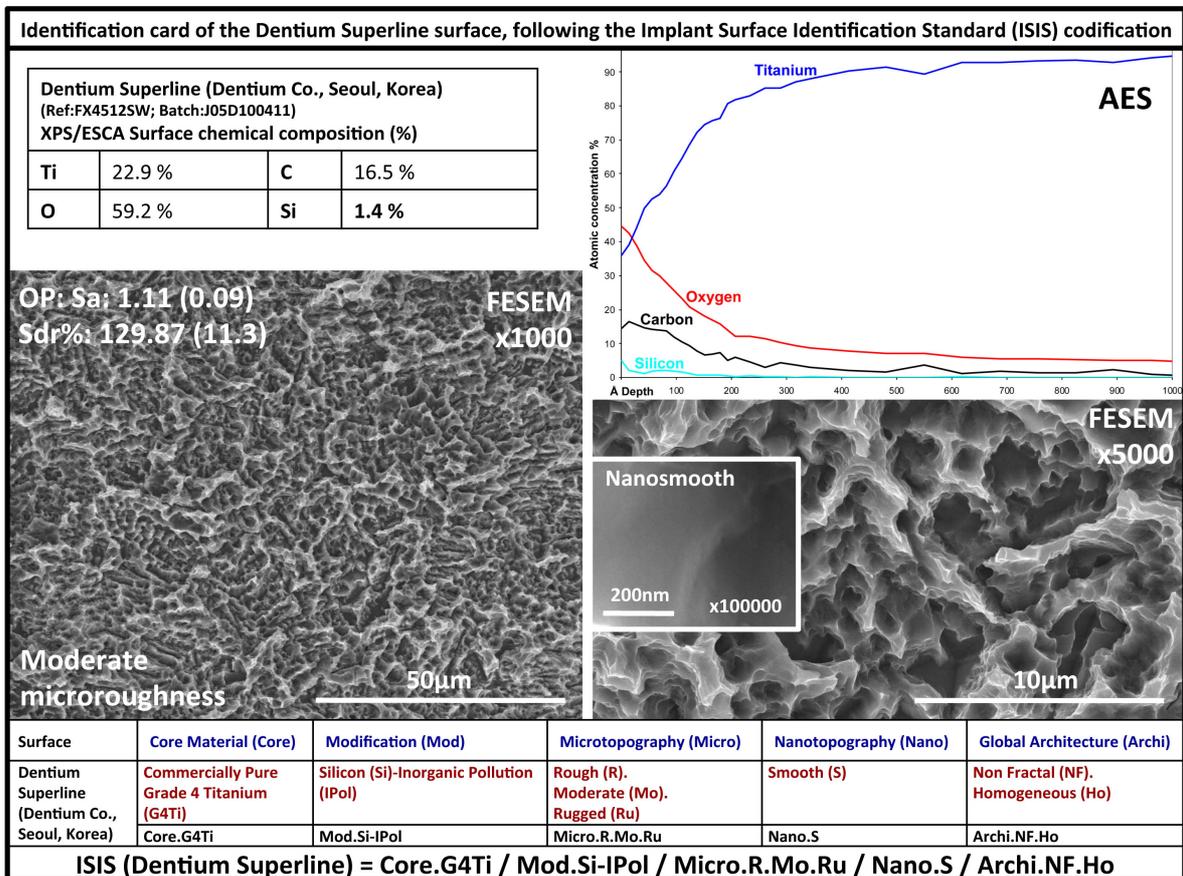


Figure 6. Identification Card of the Dentium Superline surface.

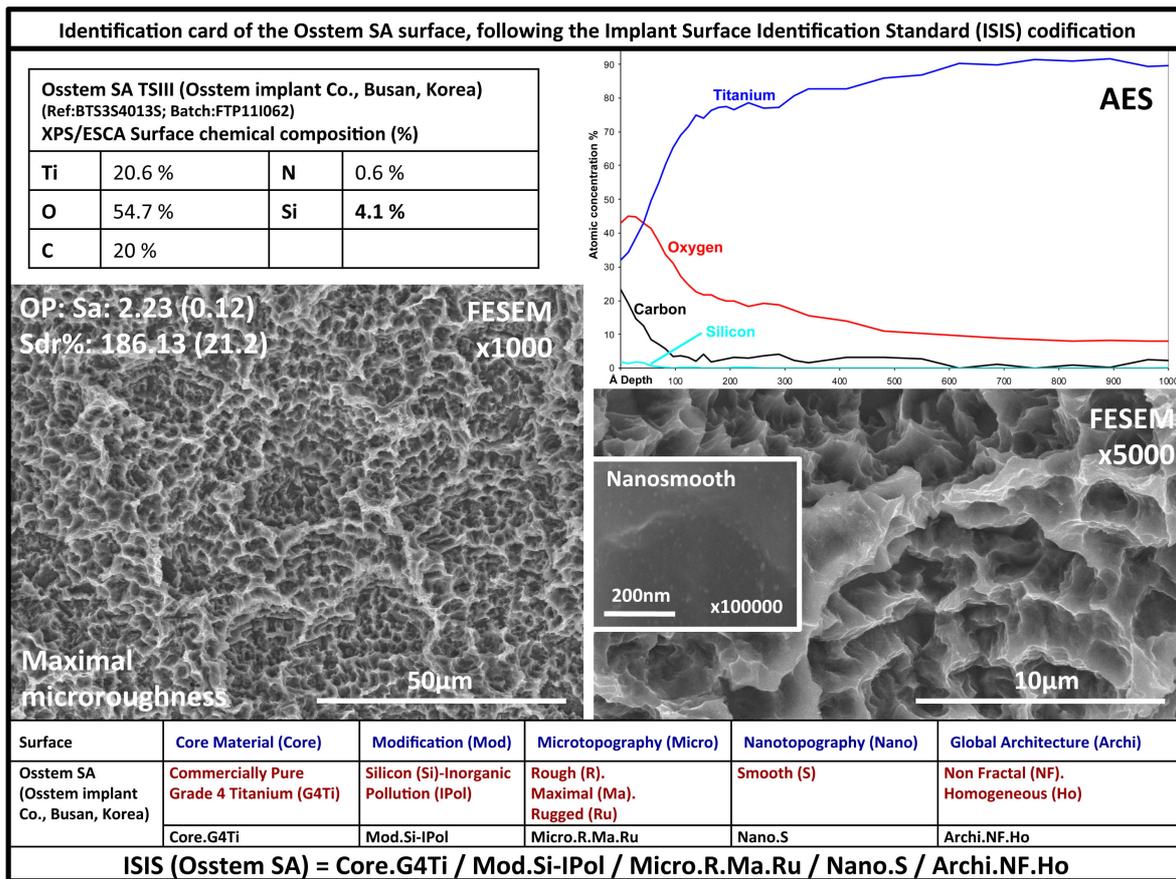


Figure 7. Identification Card of the Osstem SA surface.

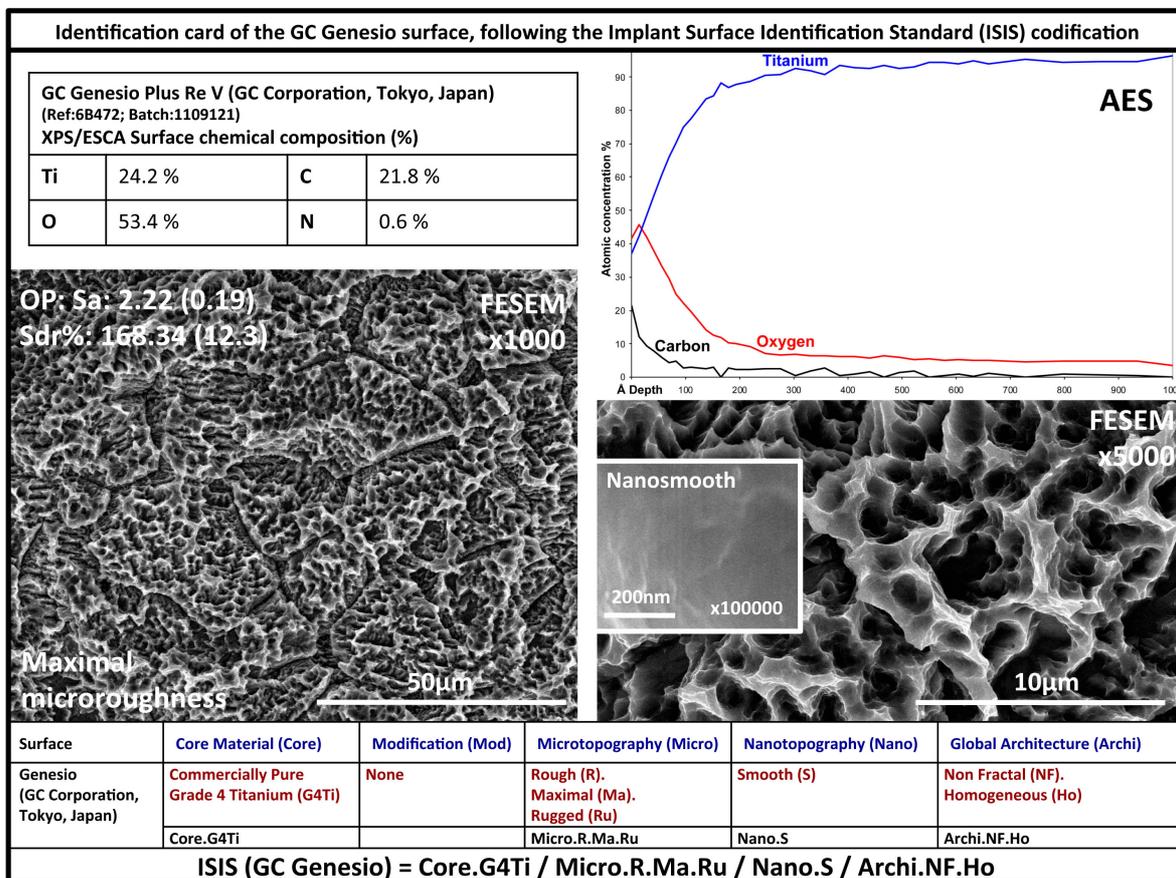


Figure 8. Identification Card of the GC Genesisio surface.

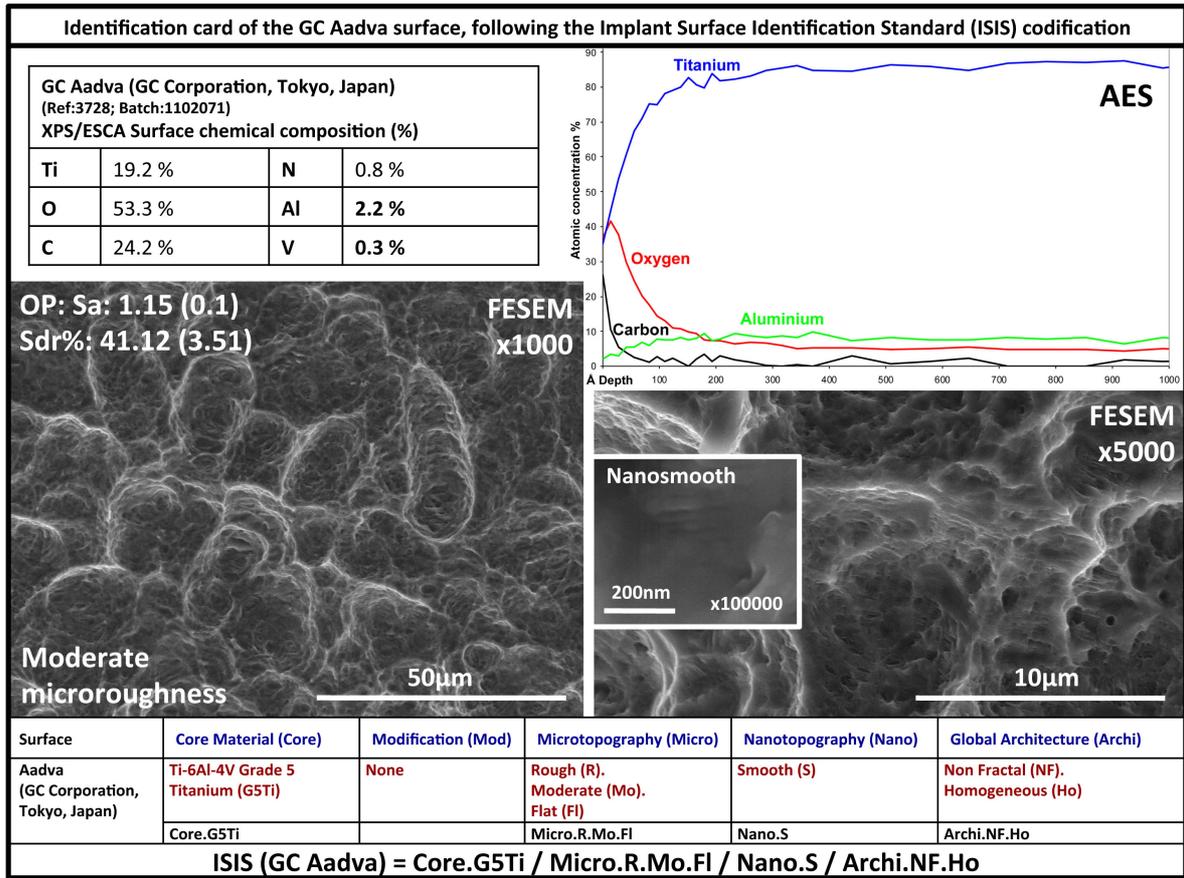


Figure 9. Identification Card of the GC Aadva surface.

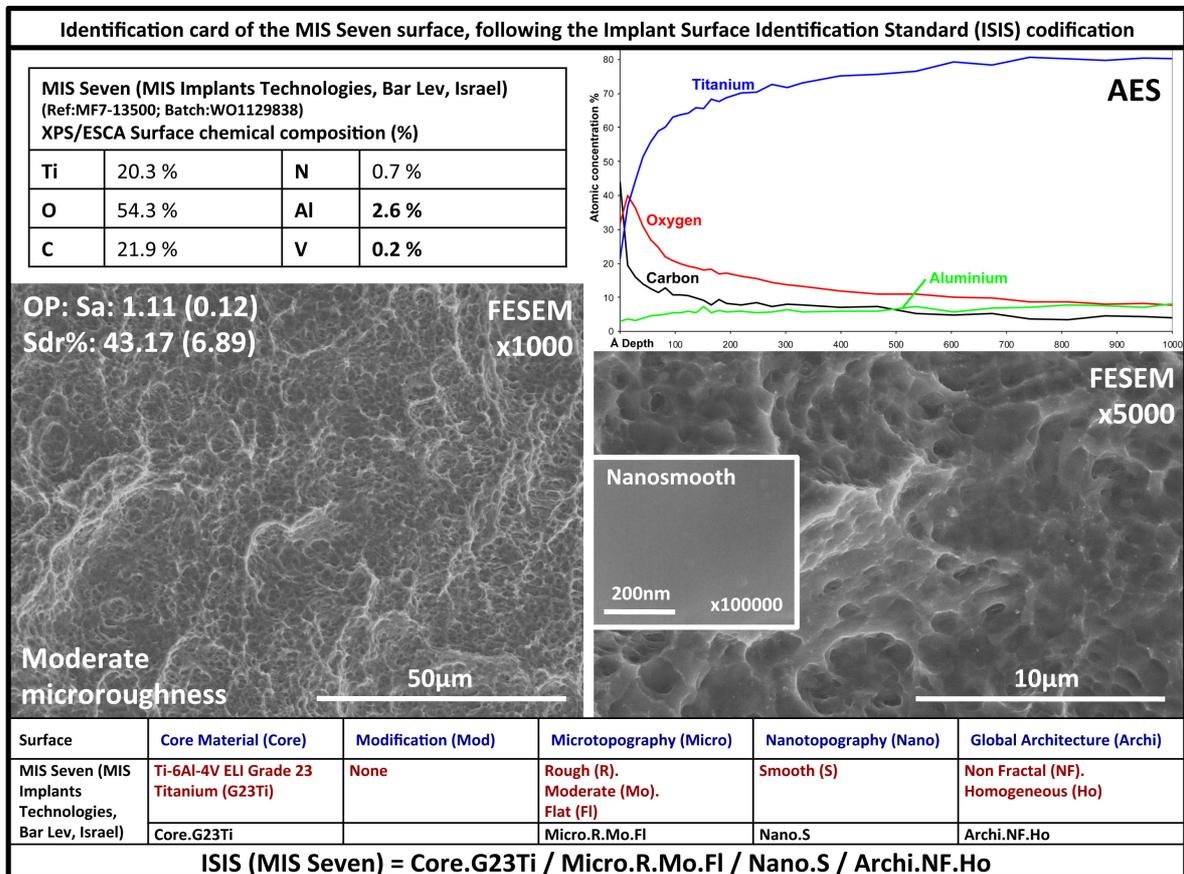


Figure 10. Identification Card of the MIS Seven surface.

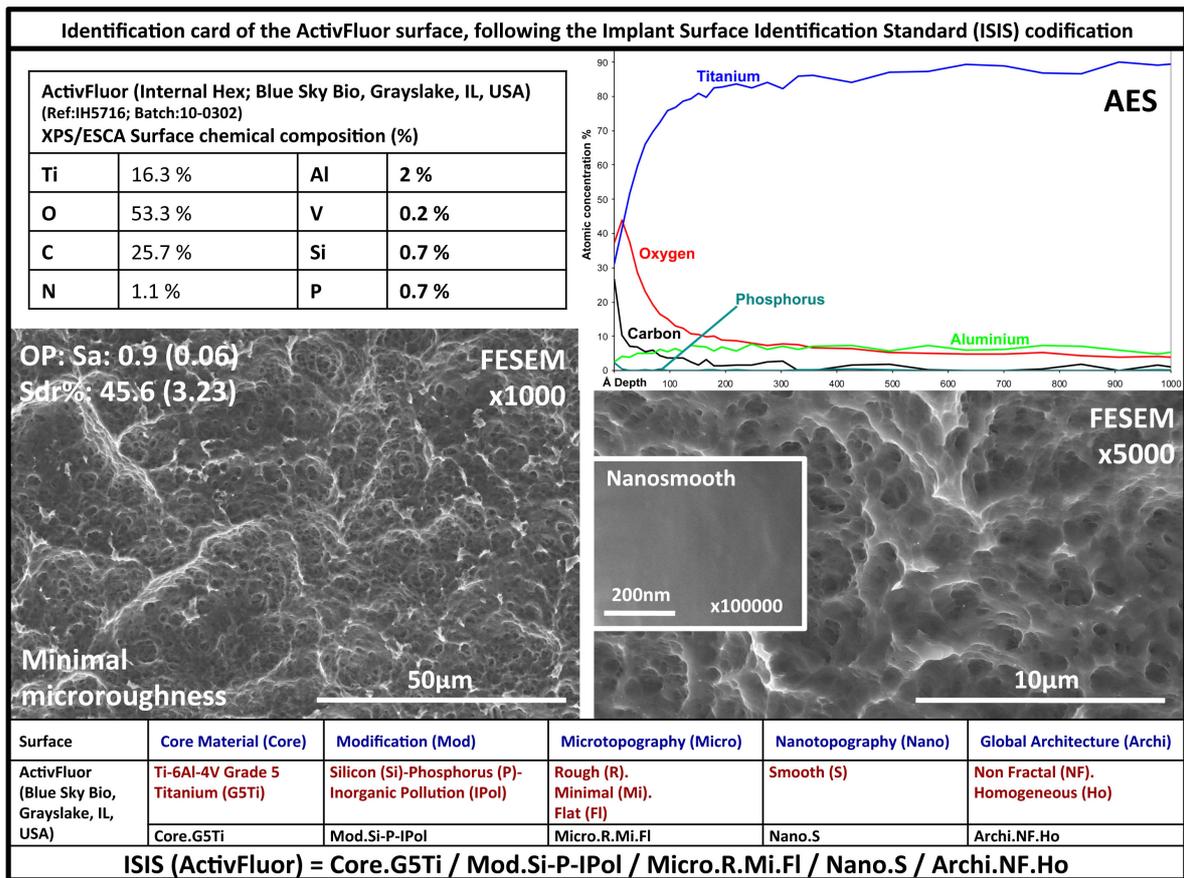


Figure 11. Identification Card of the ActivFluor surface.

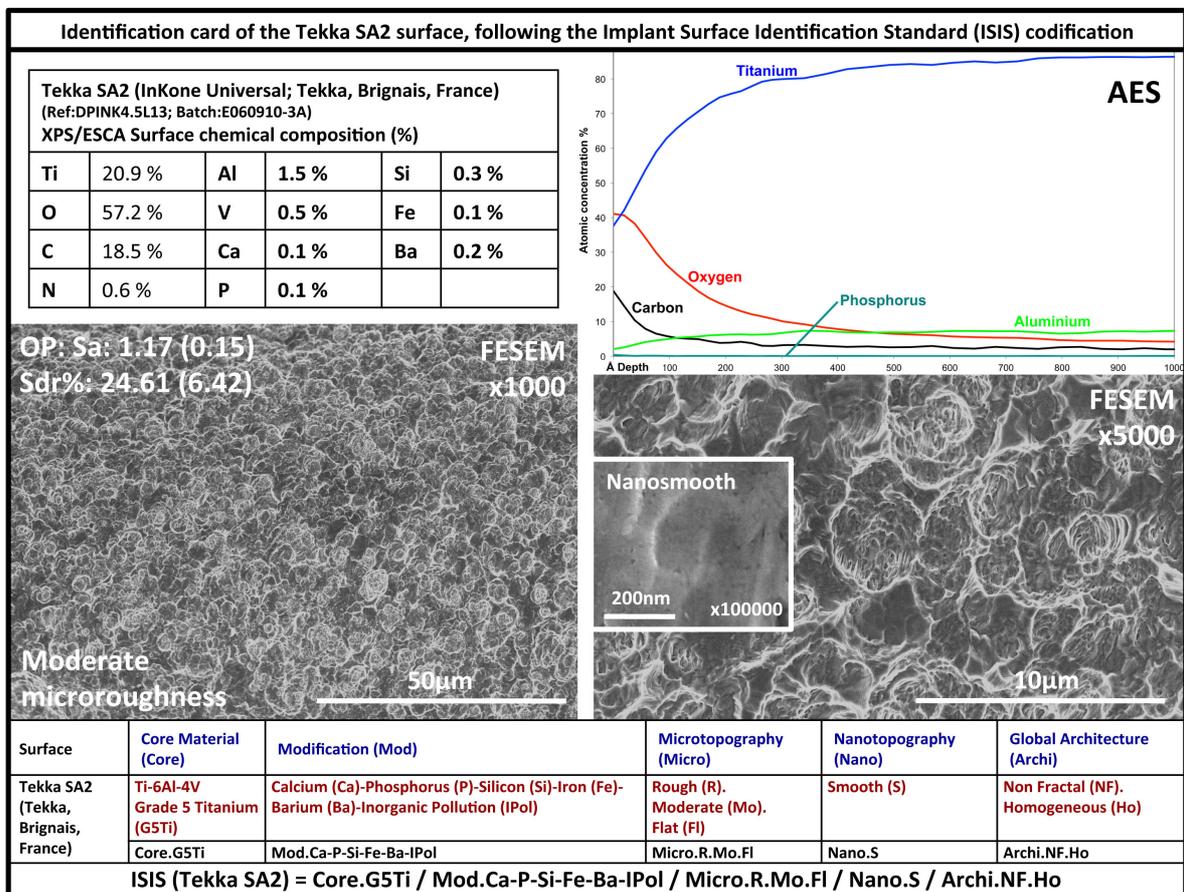


Figure 12. Identification Card of the Tekka SA2 surface.

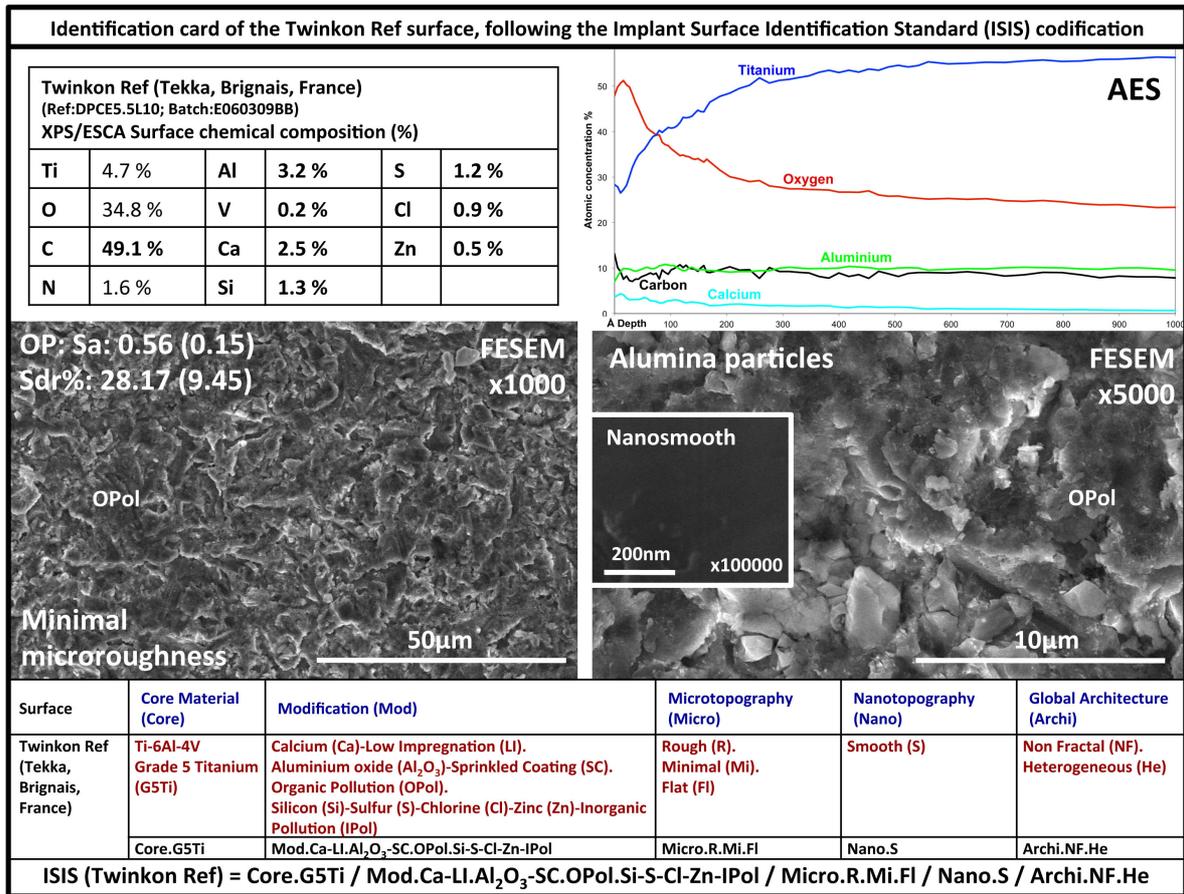


Figure 13. Identification Card of the Twinkon Ref surface.

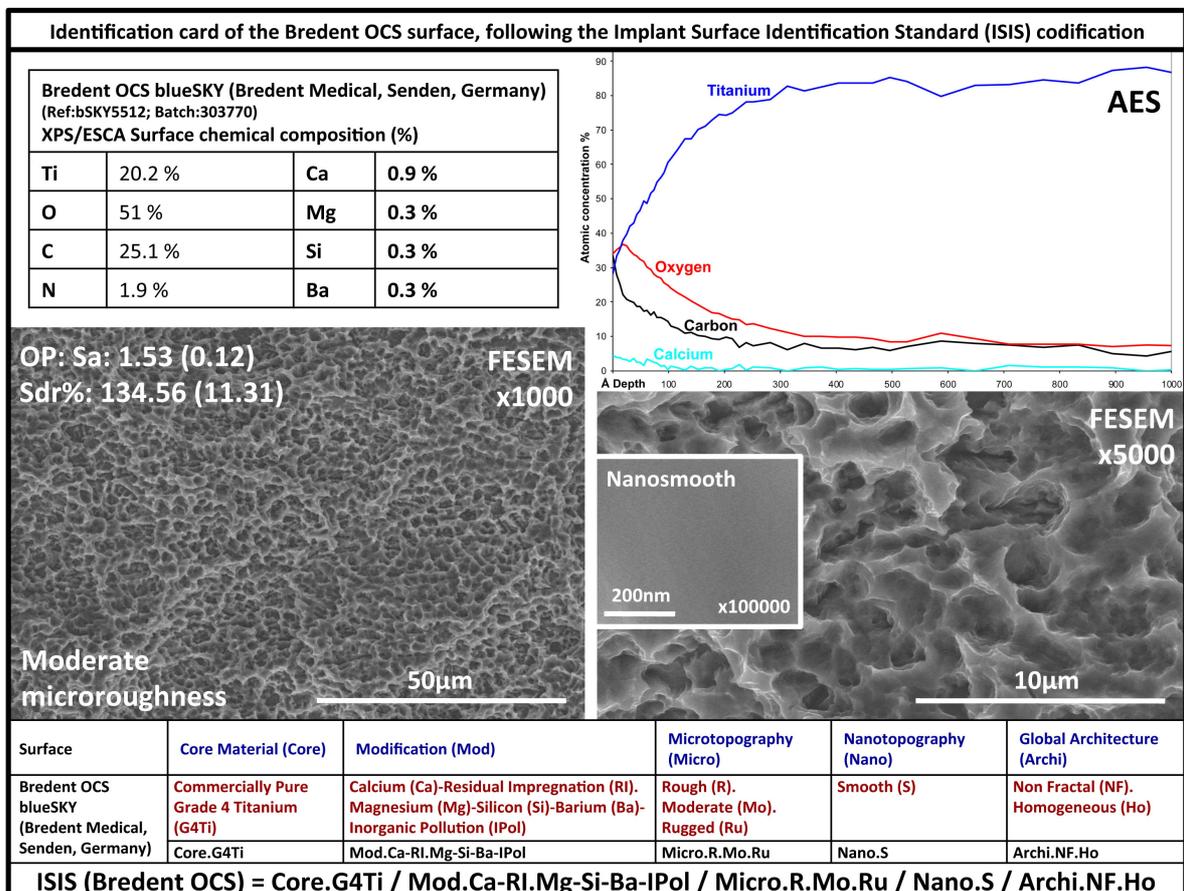


Figure 14. Identification Card of the Bredent OCS surface.

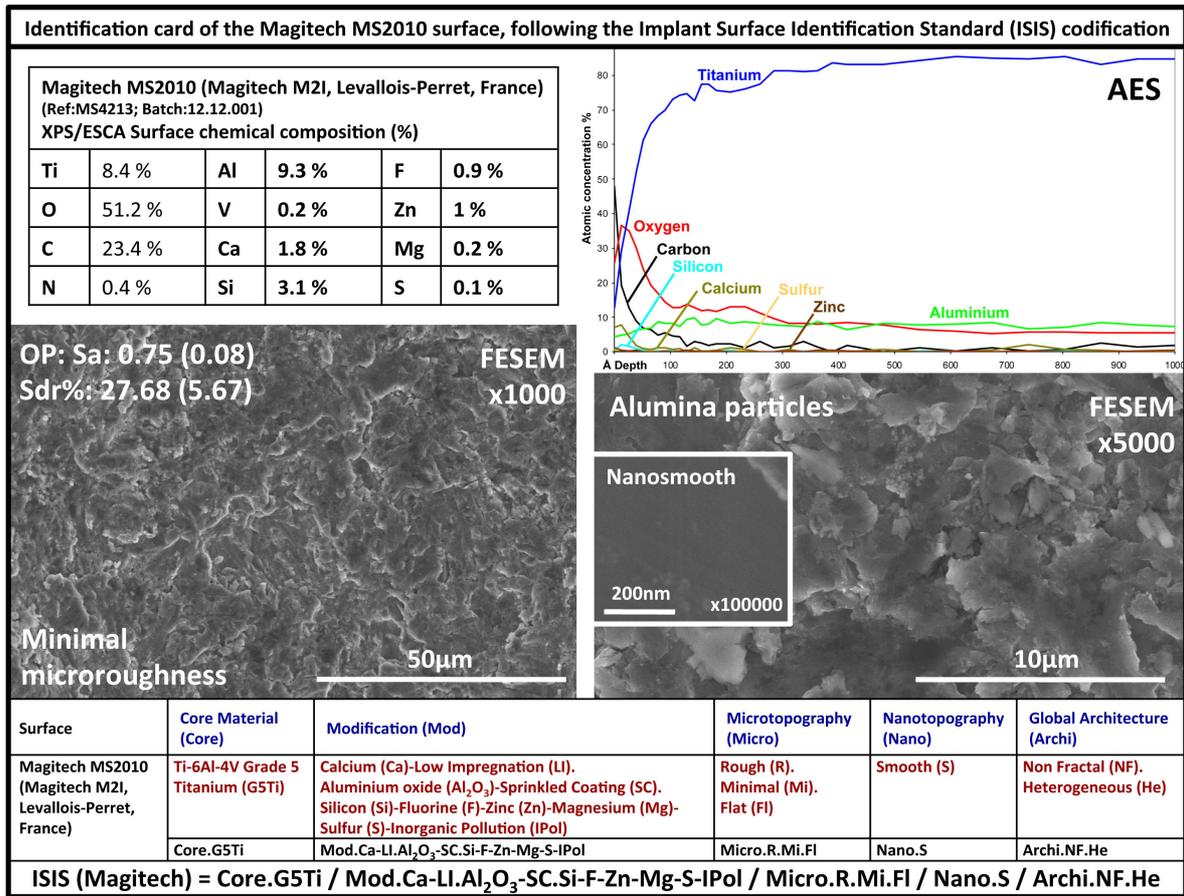


Figure 15. Identification Card of the Magitech MS2010 surface.

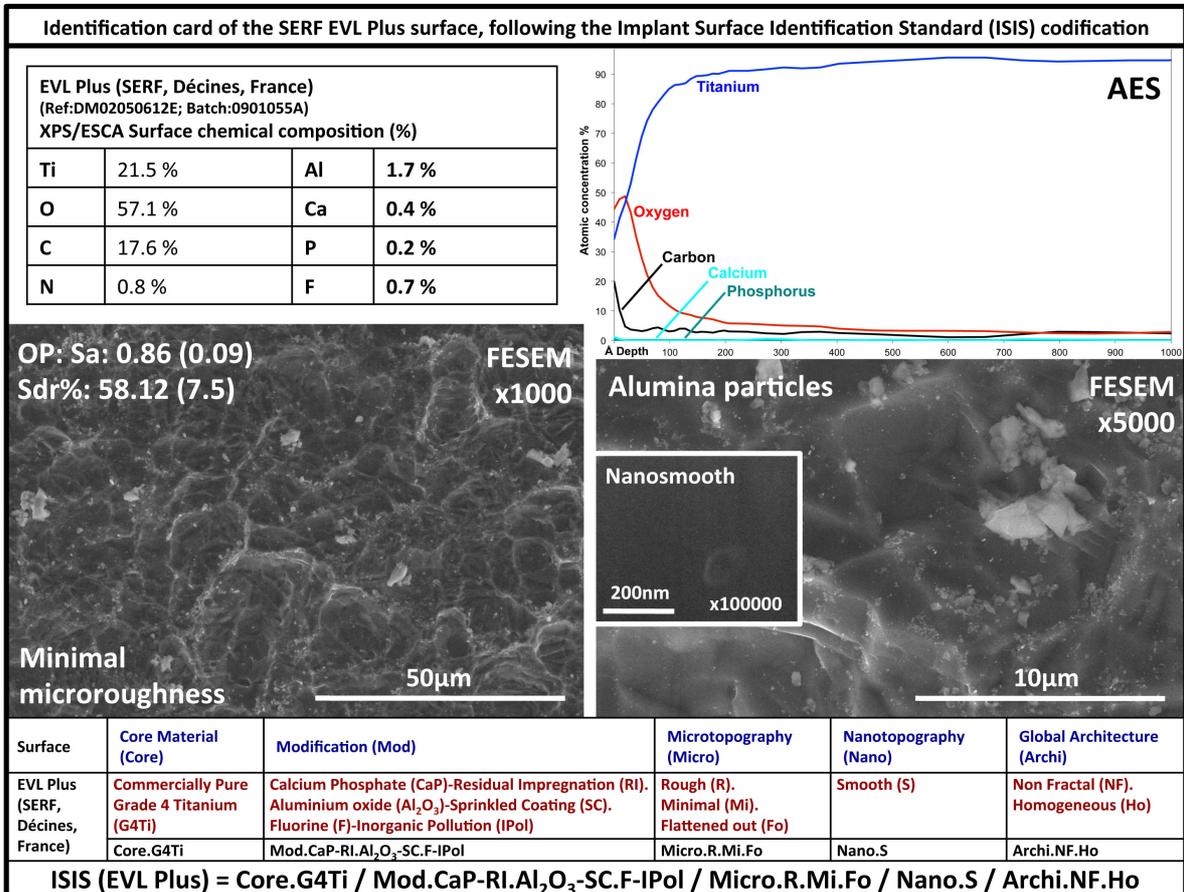


Figure 16. Identification Card of the SERF EVL Plus surface.

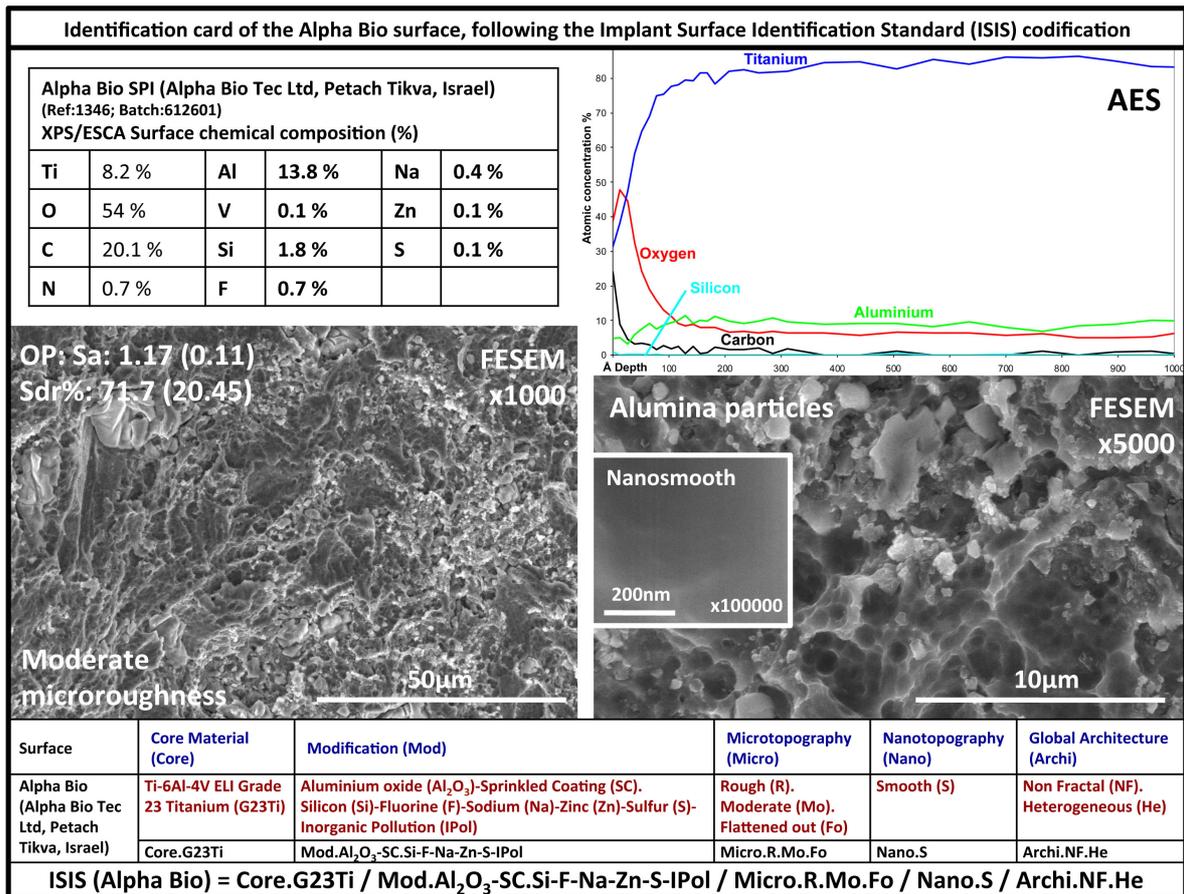


Figure 17. Identification Card of the Alpha Bio surface.

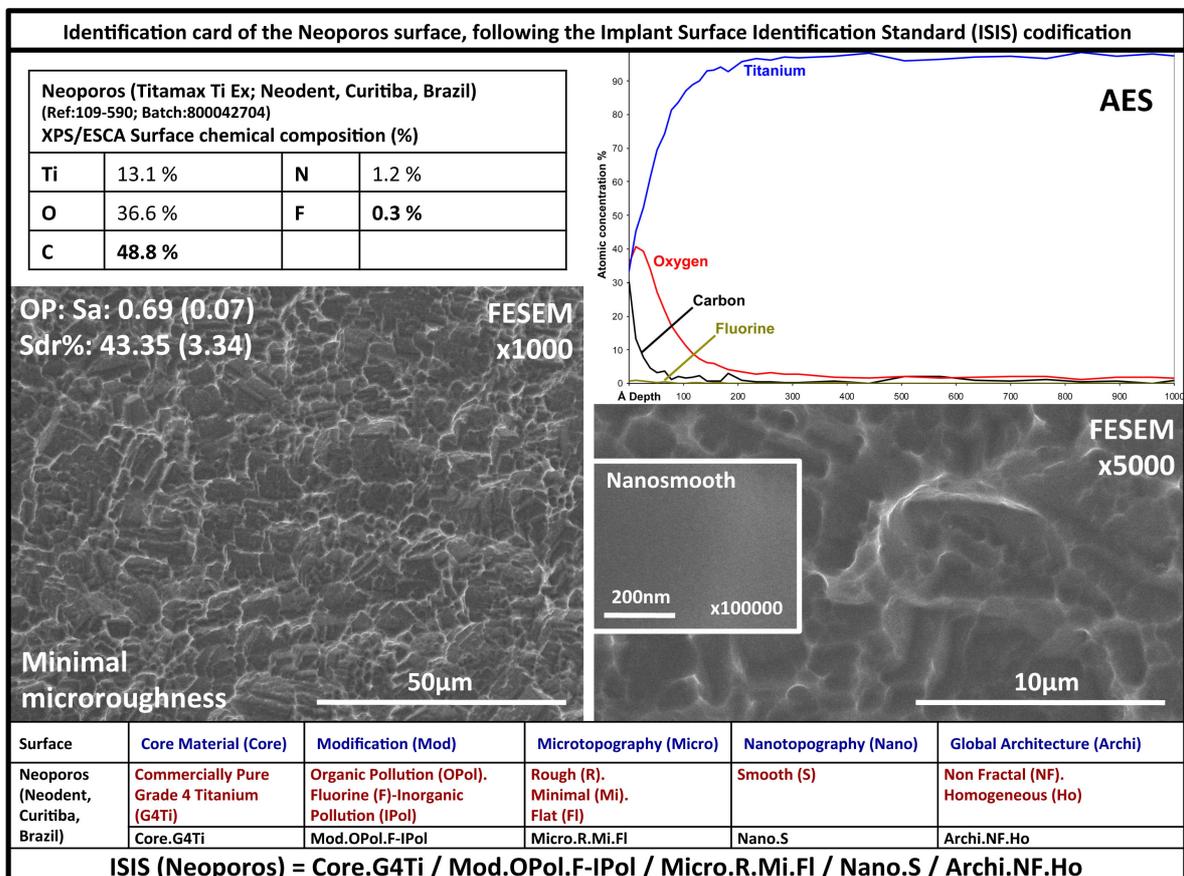


Figure 18. Identification Card of the Neoporos surface.

EVL Plus (SERF, Decines, France; **Figure 16**) was a sand-blasted/acid-etched surface. It was impregnated with residual levels of calcium phosphate and covered with small alumina (Al_2O_3) particles. A residual fluoride inorganic pollution was also detected. The surface was minimally microrough, nanosmooth, and homogeneous all over the implant.

Alpha Bio (Alpha Bio Tec Ltd, Petach Tikva, Israel; **Figure 17**) was a sand-blasted/acid-etched surface on a grade 23 ELI (Extra Low Interstitials) titanium core. It was covered with large alumina (Al_2O_3) particles. Several inorganic pollutions with silicon, fluorine, sodium, zinc and sulfur were detected. The surface was moderately microrough, nanosmooth, and heterogeneous all over the implant.

Neoporos (Neodent, Curitiba, Brazil; **Figure 18**) was a sand-blasted/acid-etched surface. The surface appeared covered with a thick organic pollution (thick carbon overcoat all over the implant). Some inorganic pollution with fluorine was also detected. The surface was minimally microrough, nanosmooth, and homogeneous all over the implant.

4. Discussion

The SLA-type surfaces represented the largest sub-group from all the implant surface technologies that were investigated in this study, revealing the very wide development of this subtractive approach in the industry. All these products have in common some characteristics, such as a microrough morphology and the systematic 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. This wide development of the method is clearly related to the good clinical results obtained with these kinds of surfaces [8,9], even if there are many debates about the ideal degree of microroughness and the risk of peri-implantitis or peri-implant bone loss [10].

A majority of implants still uses a pure titanium (grade 2 or 4) as core material, but many implant systems now use titanium alloy Ti-6Al-4V (grade 5 or grade 23 ELI - Extra Low Interstitials - titanium) as core material in order to improve the strength of the implant screws, particularly in small diameter implants. The use of titanium alloy is widely debated since many years, as these materials offer improved strength performances, but a low ductility and a different resistance to corrosion in comparison to commercially pure titanium. The debate is still opened [3]. However, the use of a different core material implies also a different calibration of the blasting/etching procedures, and companies using the 2 kinds of titanium in their production (pure titanium for standard diameter implants, alloys for smaller diameters for example) must do significant efforts of calibration to get similar surfaces on all their implants. It is also important to notice that all grade 5 or grade 23 implants of this series presented a more flattened out profile than the traditional SLA-type implants made on grade 4 or grade 2 pure titanium. A pure titanium core material is softer and in general more suitable for the engineering by subtraction of a very well defined SLA-type microtopography, while the hardness of the titanium alloys requires more efforts to carve a very elaborated microtexture.

The concept of the SLA-type surfaces is to promote a bone/implant biomechanical interlocking through the microroughness carved on the implant surfaces [1,11]. Chemical modifications are possible to stimulate the biochemical interlocking, but they are rarely advocated in the concept supporting this kind of surfaces. In this large pool of samples, four samples presented some chemical impregnation, but most of these modifications appeared as unexpected consequences from the etching process and not voluntary modification

supported by a biochemical concept. On the contrary, 15 samples were presenting various forms of inorganic pollutions related to the production process, particularly 5 surfaces were covered with Al_2O_3 alumina blasting residues. Two surfaces were presenting severe organic pollutions, which are in general considered as the contaminants with the strongest clinical impact (early implant loss or peri-implantitis)[10]. In short, in the SLA-type sub-group, chemical alterations are not expected and are mostly appearing as pollution that should be avoided. The pollution is a marker of the industrial processing standardization and control of the company, as it is very well illustrated by the example of the evolution of the Friadent surfaces: the early Ankylos surface presented many pollutions, while the latest version of this surface (used in Frialit and Xive) appeared more controlled.

In this large SLA-type family, the surfaces can be globally sorted out in 3 sub-groups. The first sub-group is gathering all the surfaces with a quite aggressive and very elaborated microtopography, based on the patterns developed in the original SLA surface: Straumann SLA, Xive S, Frialit, Camlog Promote, Dentium, Osstem SA, GC Genesio, Bredent. The second sub-group is gathering all the surfaces with a less aggressive microdesign, obtained through a different combination of material and acid-etching that smoothed a little bit the microroughness shapes: GC Aadva, MIS Seven, ActivFluor, Tekka SA2, SERF EVL Plus, Neoporos. The last sub-group gathers samples where the microtopography is not well-defined because of an incomplete industrial process (blasting residues not fully removed): early Ankylos, Tekka Twinkon Ref, Magitech MS2010, Alpha Bio. Companies of this last sub-group often did efforts in the recent years to upgrade their SLA process (as it can be observed by comparing Ankylos and Xive/Frialit), but many examples of this kind can still be found on the market.

One specific characteristic of the 18 implant surfaces of this SLA-type surface family is that none of them was presenting significant nanofeatures: all were nanosmooth, without exception. It seems therefore that the SLA-type process and its many nuances do not allow to create significant nanotexturization. It may be a technical limitation related to the method itself. However, nanofeatures were widely advocated in the recent years as the new frontier for the evolution of dental implant surfaces to improve their clinical performances [12-14]. This may explain why companies like Straumann made evolve the SLA surface through a chemical coating of the surface with Sodium Chloride NaCl crystals to produce an artificial chemical modification and nanotexture on the SLA basis (under the commercial name SLActive, placed in the Group 3 with coated surfaces)[15].

5. Conclusion

The SLA-type surfaces represent the most frequent surfaces used in the dental implant industry, and are based mostly on the concept of bone/implant biomechanical interlocking through the implant microroughness. Most surfaces of this group presented similar patterns that were easily identifiable. 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. The development of this surface was associated with good clinical results in comparison to other potential surfaces, what is probably the reason of its frequent use. In the recent years, companies tried to improve this technology by reducing contaminants and adding some chemical modification and nanofeatures on the SLA-type basis. It is expected that this approach will develop in the coming years.

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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