

Research article

Identification card and codification of the chemical and morphological characteristics of 62 dental implant surfaces. Part 2: anodized and Titanium Plasma-Sprayed (TPS) surfaces (Group 1, metallurgy modification)

David M. Dohan Ehrenfest,^{1,2,*} Marco Del Corso,^{3,4} Byung-Soo Kang,⁵ Philippe Leclercq,⁶ Ziv Mazor,⁷ Robert A. Horowitz,⁸ Philippe Russe,⁹ Hee-Kyun Oh,¹⁰ De-Rong Zou,¹¹ Jamil Awad Shibli,¹² Hom-Lay Wang,¹³ Jean-Pierre Bernard,² and Gilberto Sammartino.³

¹ LoB5 unit, Research Center for Biomineralization Disorders, Chonnam National University, South Korea

² Department of Stomatology, School of Dental Medicine, University of Geneva, Switzerland

³ Department of Oral Surgery, Faculty of Medicine, University Federico II of Naples, Italy

⁴ Private Practice, Turin, Italy

⁵ Department of Physics, Seoul National University, Seoul, South Korea

⁶ Private Practice, Paris, France

⁷ Private Practice, Ra'anana, Israel

⁸ Department of Periodontology and Implant Dentistry, College of Dentistry, New York University, New York, USA

⁹ Private Practice, Reims, France

¹⁰ Department of Oral and Maxillofacial Surgery, School of Dentistry, Chonnam National University, South Korea

¹¹ Department of Stomatology, Shanghai Sixth People's Hospital, Shanghai Jiao Tong University, China

¹² Department of Periodontology and Oral Implantology, University of Guarulhos, Sao Paulo, Brazil

¹³ Department of Periodontics and Oral Medicine, School of Dentistry, University of Michigan, Ann Arbor, USA

*Corresponding author: David M. Dohan Ehrenfest, LoB5@mac.com

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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 second part, surfaces with metallurgy modification (anodization, titanium plasma-spraying TPS) were investigated.

Materials and Methods. Eight different implant surfaces were characterized: TiUnite (Nobel Biocare, Gothenburg, Sweden), Ospol (Ospol, Höllviken, Sweden), INNO (Cowellmedi Co., Busan, Korea), Shinhung M (Shinhung Co., Seoul, Korea), Tecom REP (Tecom Implantology/Titanmed, Galbiate, Italy), BioSpark (Keystone Dental, Burlington, MA, USA), Kohno HRPS (Sweden & Martina, Due Carrare, Italy), Kohno DES HRPS (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 8 different surfaces of this group, all were based on a commercially pure titanium (grade 2 or 4), what appeared typical of surfaces produced through a modification of the core material metallurgy using anodization or titanium-plasma spraying. The 6 anodized surfaces presented different forms of chemical impregnation of the titanium core. Seven surfaces presented different degrees of inorganic pollutions. Only 1 surface presented no pollution. From a morphological standpoint, 5 surfaces were microporous (anodization) and 3 microrough, with different microtopographical aspects and values. Seven surfaces were smooth on the nanoscale, and therefore presented no significant and repetitive nanostructures. One implant was nanopatterned through a specific anodization process. Six implants presented various forms of cracks: three anodized implants had local cracks, while TiUnite and Kohnno HRPS were covered with extended cracks all over the surface. Anodized surfaces could be considered as homogeneous, while TPS surfaces were heterogeneous (specificities of the production process). No surface 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 implants of the Group 1 have very specific morphological characteristics (frequent cracks and absence of nanotexture, specific microroughness or porosity), and users should be aware of these specificities if they decide to use these specific technologies.

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]. 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 [3,4]. 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 [5,6]. 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 [5,7]. 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 [5]. This final system, termed ISIS (Implant Surface Identification Standard) may be used as an official international standard in the future.

The first category of methods (arbitrarily termed Group 1) to create a dental implant surface is to modify the characteristics of the core material during the process. In this category, the most frequent method nowadays is the titanium anodization, i.e. an electrochemical process where the implant is placed in an electrolytic solution while electric current is applied [8]. The concept is to provoke an in-depth impregnation of the titanium external layer with specific ions (calcium, phosphorus or magnesium in general)[9] and the

creation of a very thick (several micrometers) layer of TiO_2 all around the implant, both characteristics expected to improve the osseointegration through a better mineral nucleation [10]. The other consequences of these techniques are the development of specific morphological patterns, in general a significant microporosity [11], even if many micro- or nano-patterns are possible [12]. The concept of anodization is often associated with the TiUnite surface (Nobel Biocare), as it is the largest company promoting this technology. In the Group 1, the second classical method is the use of titanium plasma-spraying (TPS), in order to cover the surface with a very aggressive microroughness [13].

In this second part, the chemical and morphological characteristics of 8 implant surfaces (available on the market) from the first group were investigated and described through a simple and clear identification (ID) card for each surface, following the ISIS system terminology and classification. The first group gathered all surfaces produced through modification of the core material characteristics, mostly the alteration of the titanium metallurgy through anodization or titanium-plasma spraying (TPS).

2. Materials and Methods

2.1. Samples

Eight different implant surfaces of the Group 1 have been investigated: TiUnite (Nobel Biocare, Gothenburg, Sweden), Ospol (Ospol, Höllviken, Sweden), INNO (Cowellmedi Co., Busan, Korea), Shinhung M (Shinhung Co., Seoul, Korea), Tecom REP (Tecom Implantology/Titanmed, Galbiate, Italy), BioSpark (Keystone Dental, Burlington, MA, USA), Kohno HRPS (Sweden & Martina, Due Carrare, Italy), Kohno DES HRPS (Sweden & Martina, Due Carrare, 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.

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 μm 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)[5]. 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 [5], 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 8 different surfaces of this group, all were based on a commercially pure titanium (grade 2 or 4), what appeared typical of surfaces produced through a modification of the core material metallurgy using anodization or titanium-plasma spraying. The 6 anodized surfaces presented different forms of chemical impregnation of the titanium core. Seven surfaces presented different degrees of inorganic pollutions. Only 1 surface presented no pollution.

From a morphological standpoint, 5 surfaces were microporous (anodization) and 3 microrough, with different microtopographical aspects and values. Seven surfaces were smooth on the nanoscale, and therefore presented no significant and repetitive nanostructures. One implant was nanopatterned through a specific anodization process. Six implants presented various forms of cracks: three anodized implants had local cracks, while TiUnite and Kohno HRPS were covered with extended cracks all over the surface. Anodized surfaces could be considered as homogeneous, while TPS surfaces were heterogeneous (specificities of the production process). No surface 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. Anodized surfaces

The 6 first surfaces were anodized and were all chemically impregnated with thick TiO_2 superficial layers. Inorganic pollution was often detected. Five surfaces had in common to be microporous (with different degrees of porosity) and nanosmooth, and 4 presented various forms of cracks. One surface only was microrough and nanopatterned.

TiUnite (Nobel Biocare, Gothenburg, Sweden; **Figure 1**) was an anodized surface, thus presenting a thick TiO_2 layer ($>100\text{nm}$). During anodization, a high quantity of phosphorus was incorporated into the surface as a chemical modification. Inorganic pollutions with fluoride and sulfate were also detected. The surface was microporous (pores created by anodization), smooth on the nanoscale and presented extended cracks related to the anodization process.

Ospol (Ospol, Höllviken, Sweden; **Figure 2**) was an anodized surface, thus presenting a thick TiO_2 layer ($>100\text{nm}$). During anodization, low quantities of calcium and phosphorus were incorporated into the surface as a chemical modification. Traces of sodium were also detected. The surface was microporous (pores created by anodization), smooth on the nanoscale and presented small local cracks related to the anodization process.

INNO (Cowellmedi Co., Busan, Korea; **Figure 3**) was an anodized surface, thus presenting a thick TiO_2 layer ($>100\text{nm}$). During anodization, a high quantity of phosphorus (and a residual quantity of calcium) was incorporated into the surface as a chemical modification. The surface was microporous (pores created by anodization), smooth on the nanoscale and presented small local cracks related to the anodization process.

Shinhung M (Shinhung Co., Seoul, Korea; **Figure 4**) was an anodized surface, thus presenting a thick TiO_2 layer ($>100\text{nm}$). During anodization, low quantities of phosphorus and magnesium were incorporated into the surface as a chemical modification. Inorganic pollutions with silicon and sulfate were also detected. The surface was microporous (pores

created by anodization), smooth on the nanoscale and presented small local cracks related to the anodization process.

Tecom REP (Tecom Implantology/Titanmed, Galbiate, Italy; **Figure 5**) was an anodized surface, thus presenting a thick TiO₂ layer (>100nm). During anodization, a high quantity of phosphorus was incorporated into the surface as a chemical modification. Inorganic pollutions with sulfur, silicon and magnesium were also detected. The surface was microporous (pores created by anodization) and smooth on the nanoscale.

BioSpark (Keystone Dental, Burlington, MA, USA; **Figure 6**) was an anodized surface, thus presenting a thick TiO₂ layer (>100nm). During anodization, low quantities of calcium and phosphorus were incorporated into the surface as a chemical modification. Inorganic pollutions with silicon and fluorine were also detected. The surface was microrough (roughness created before anodization) and patterned at the nanoscale (the nanopatterning was shaped like nets and was done through the specific anodization process).

3.3. Titanium Plasma-Sprayed TPS surfaces

Only 2 TPS surfaces were analyzed. Both presented inorganic pollutions. Surfaces of this type had in common to be maximally rough, nanosmooth, heterogeneous and to present extended cracks all over the surface.

Kohno HRPS (High Roughness Plasma Spray; Sweden & Martina, Due Carrare, Italy; **Figure 7**) was a titanium plasma-sprayed (TPS) surface. Some inorganic pollutions were detected with phosphorus (as phosphate), fluorine and sulfur (as sulfate). The main characteristics of this kind of surfaces were topographical: the microroughness was maximal, heterogeneous, smooth on the nanoscale, and covered with many extended cracks (related to the cooling of the plasma-sprayed titanium).

Kohno DES HRPS (High Roughness Plasma Spray; Sweden & Martina, Due Carrare, Italy; **Figure 8**) was the latest version of the TPS surface of this company. The particularity of this implant DES (Dual Engineered Surface) was the presence of 2 surfaces: TPS for the 2/3 of the implant from the apex for bone/implant interface, and a blasted/etched surface (ZirTi) with much smaller roughness for the cervical area (to reduce the risk of peri-implant cervical bone loss). Here we only analyzed the TPS surface part of the implant (cervical part was analyzed in the part 5 of this series of articles). Some inorganic pollutions were detected with phosphorus (as phosphate), silicon and sulfur (as sulfate). The main characteristics of this kind of surfaces were topographical: the microroughness was maximal, heterogeneous, smooth on the nanoscale, and covered with many extended cracks (related to the cooling of the plasma-sprayed titanium).

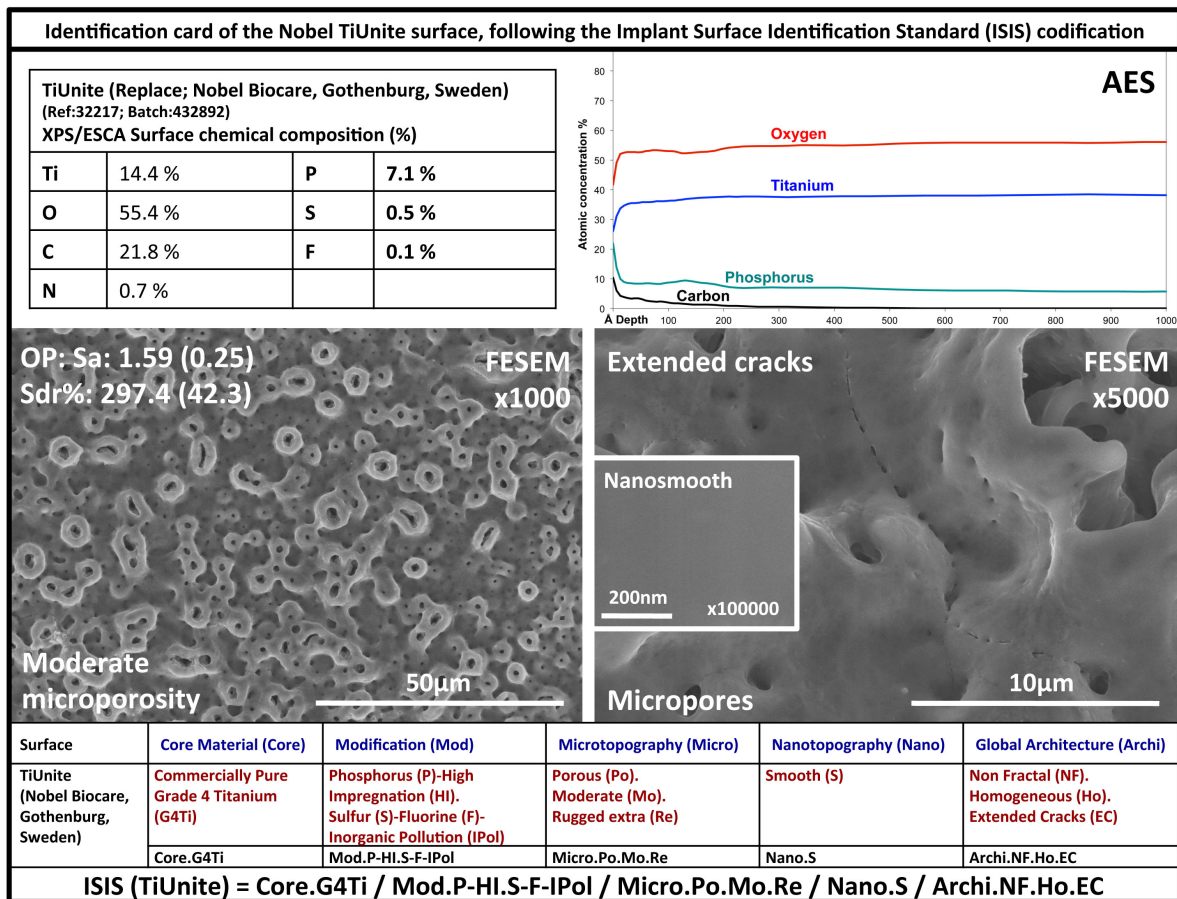


Figure 1. Identification Card of the TiUnite surface.

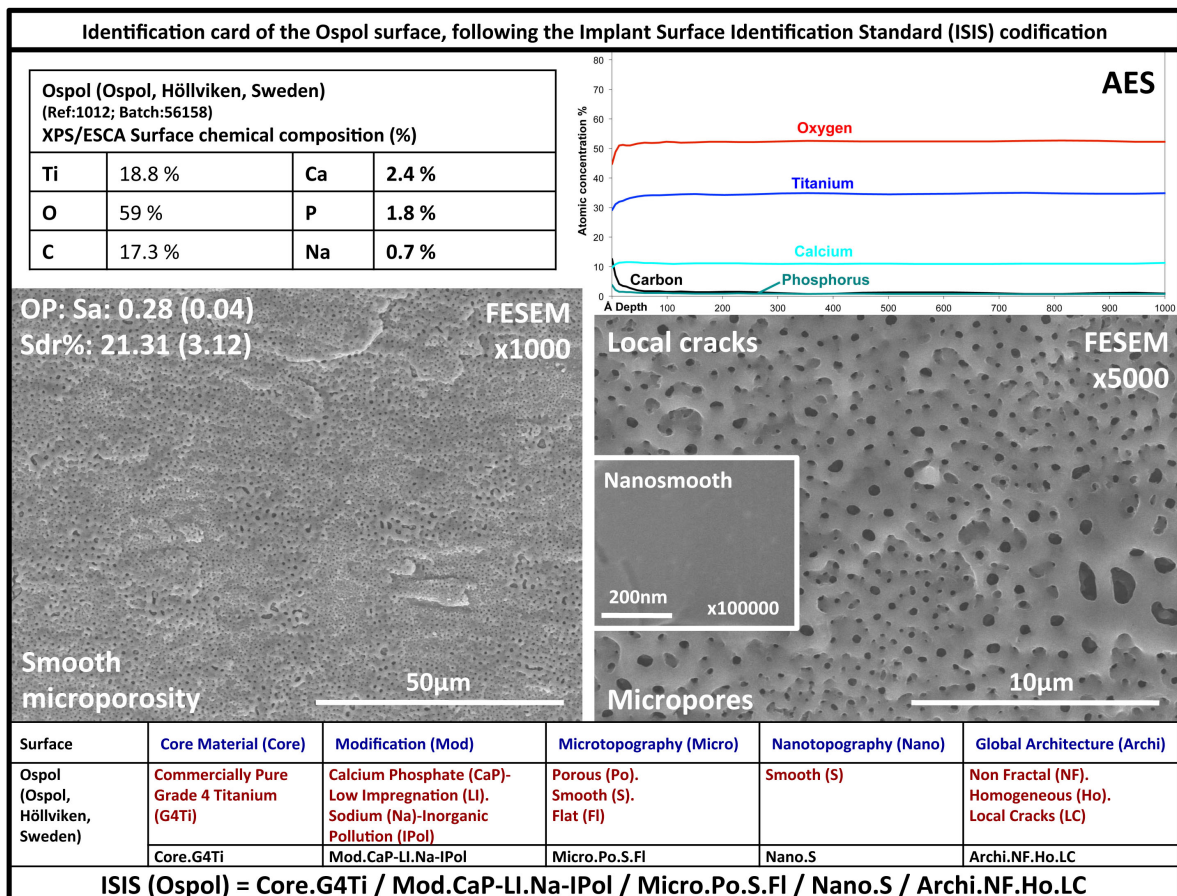


Figure 2. Identification Card of the Ospol surface.

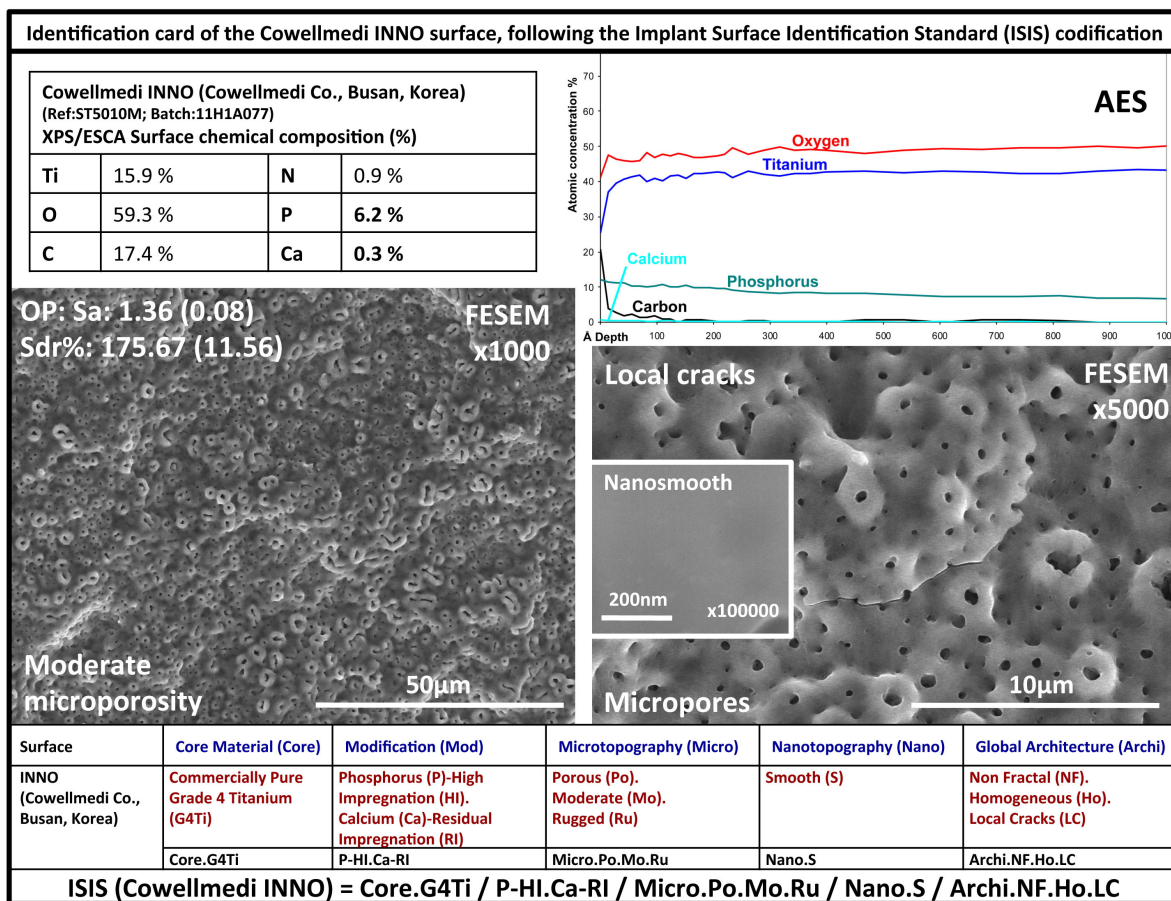


Figure 3. Identification Card of the INNO surface.

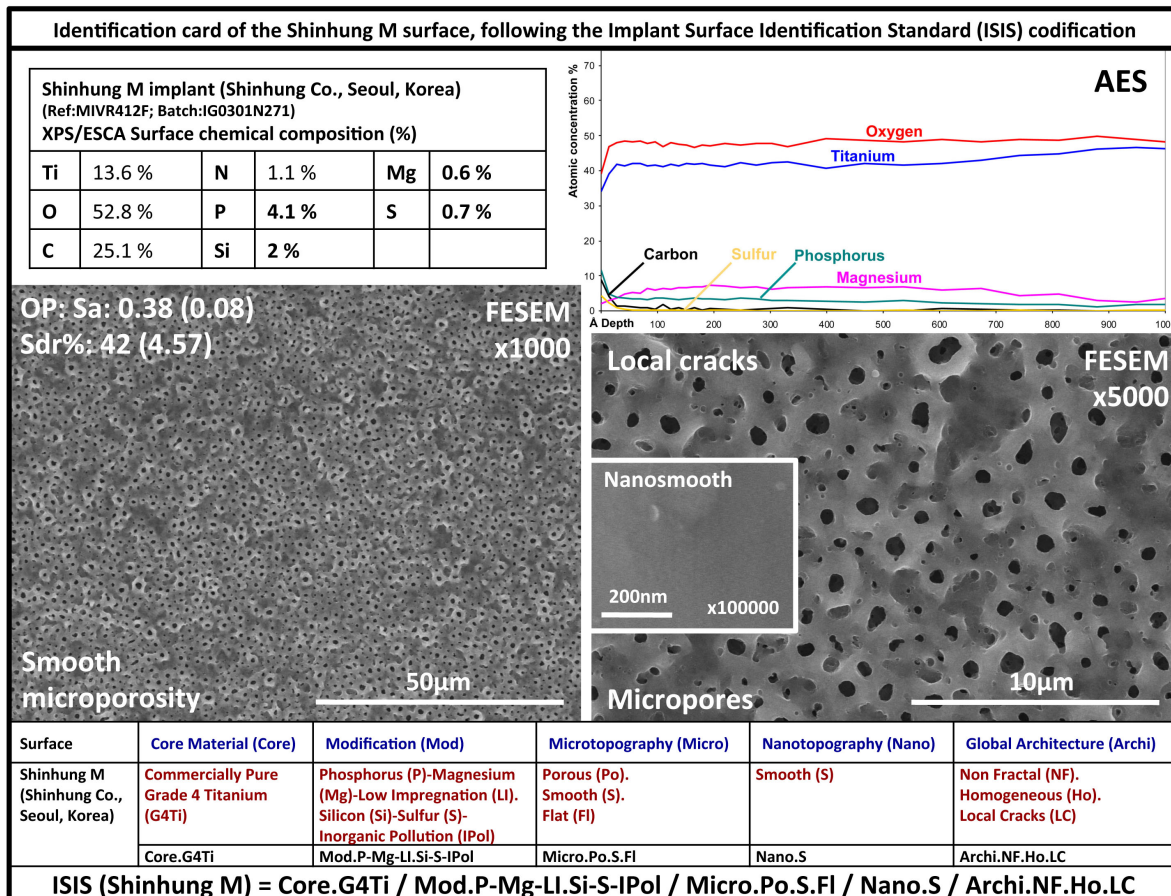


Figure 4. Identification Card of the Shinhung M surface.

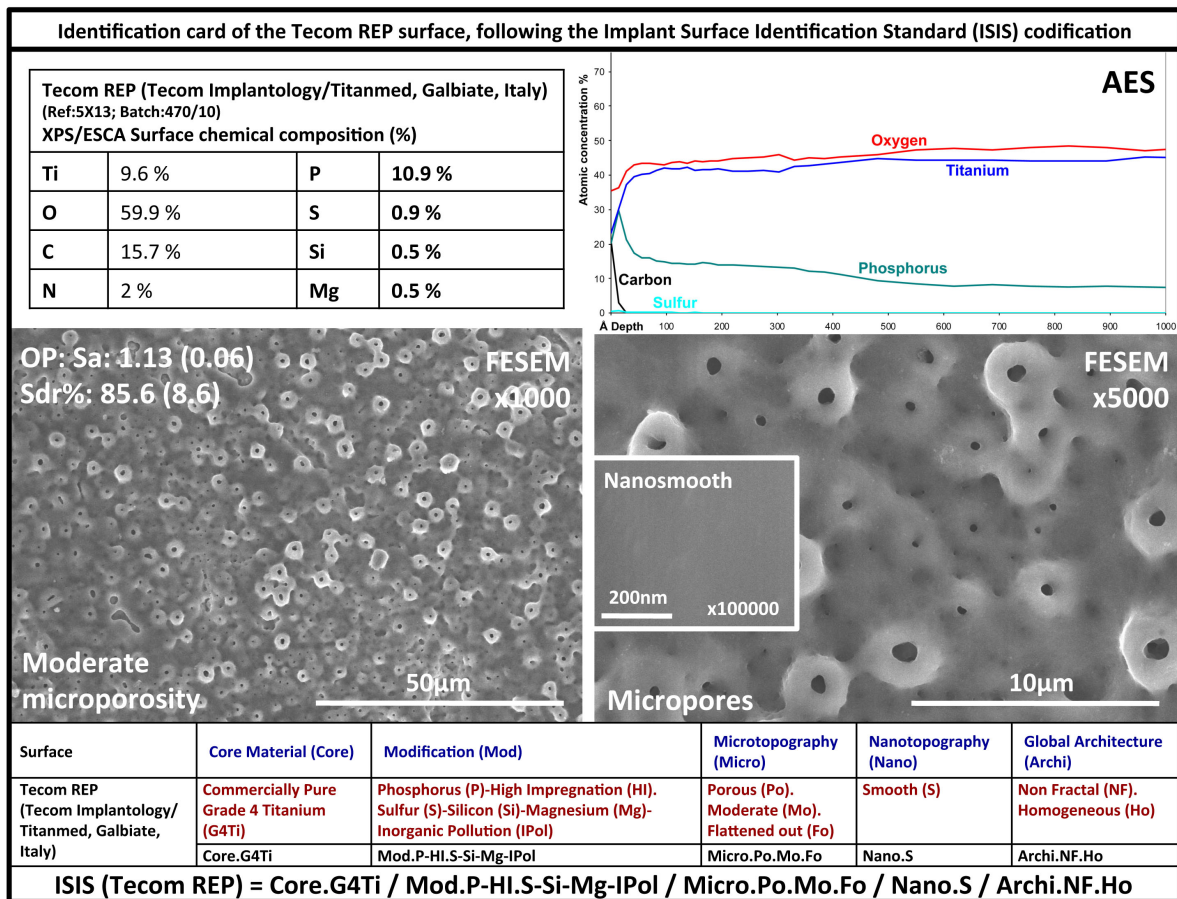


Figure 5. Identification Card of the Tecom REP surface.

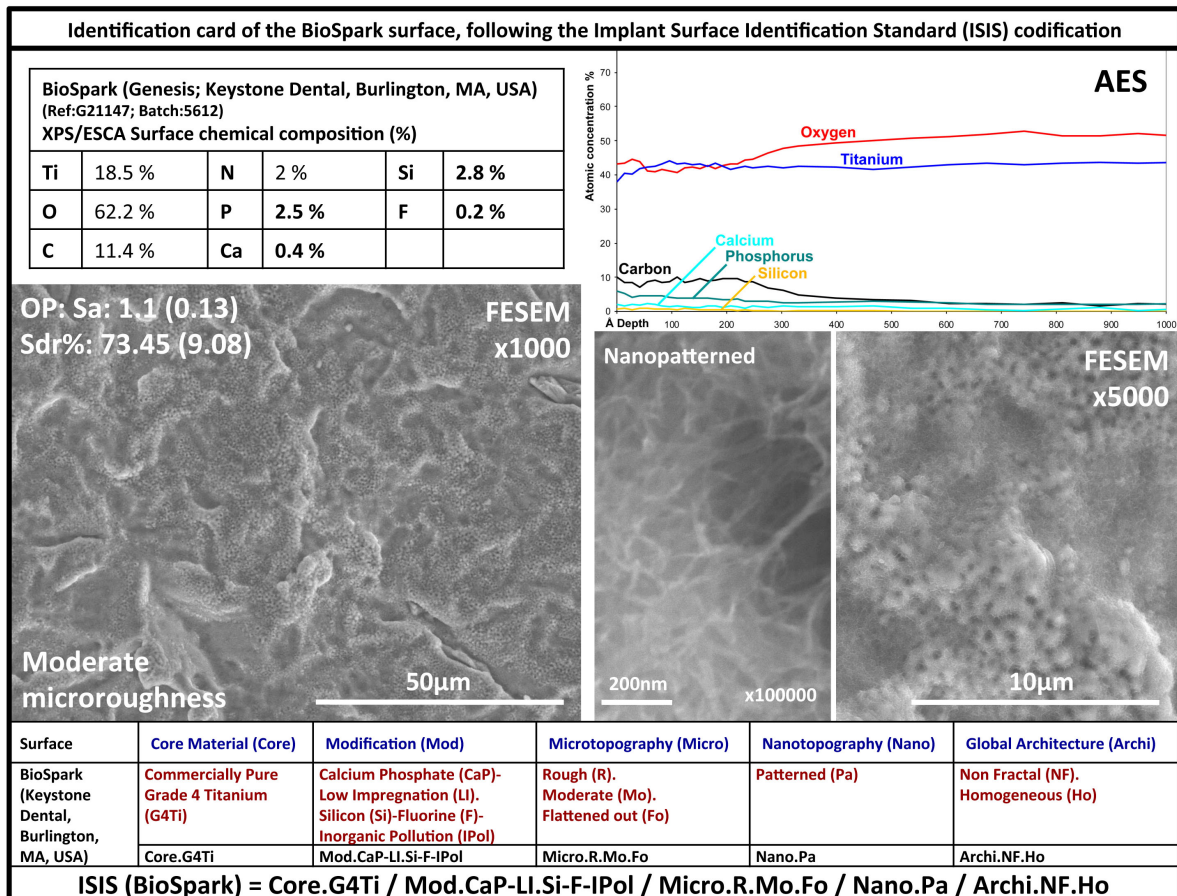


Figure 6. Identification Card of the BioSpark surface.

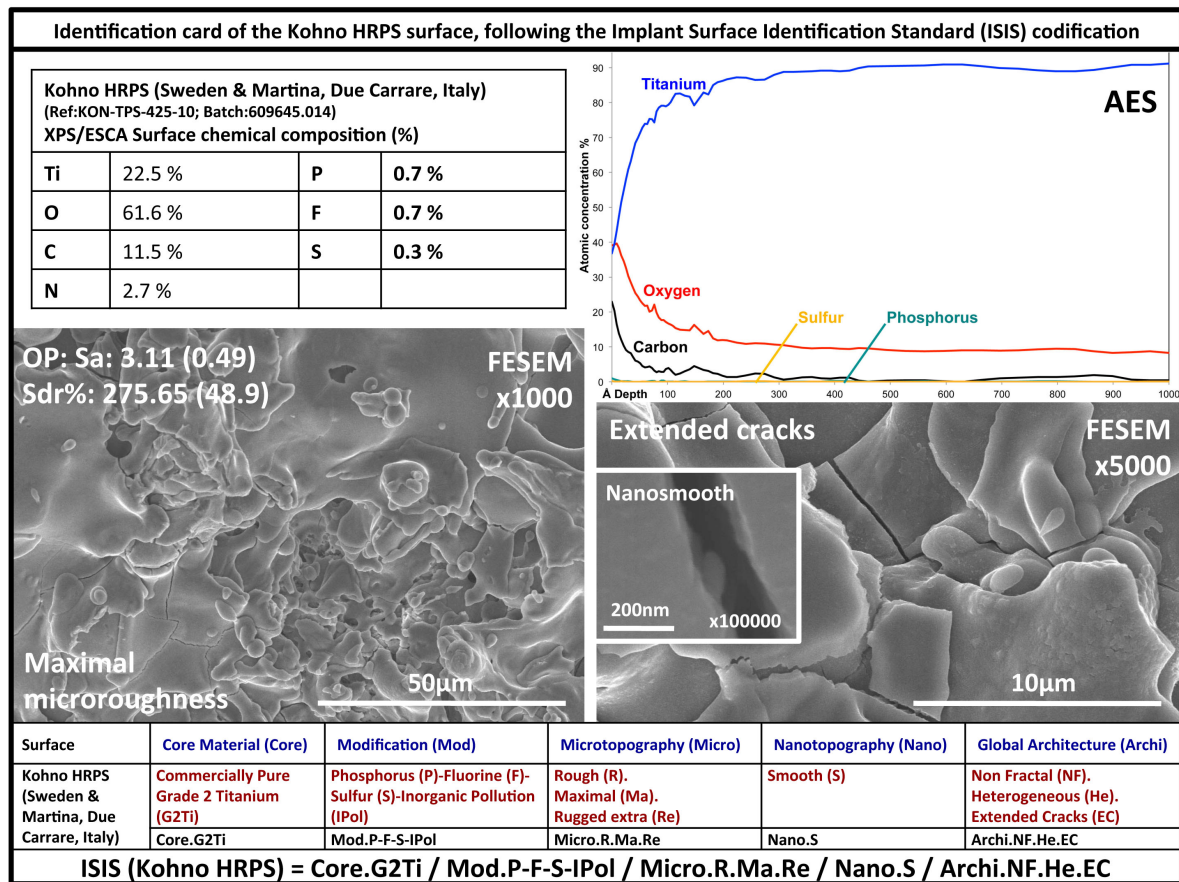


Figure 7. Identification Card of the Kohno HRPS surface.

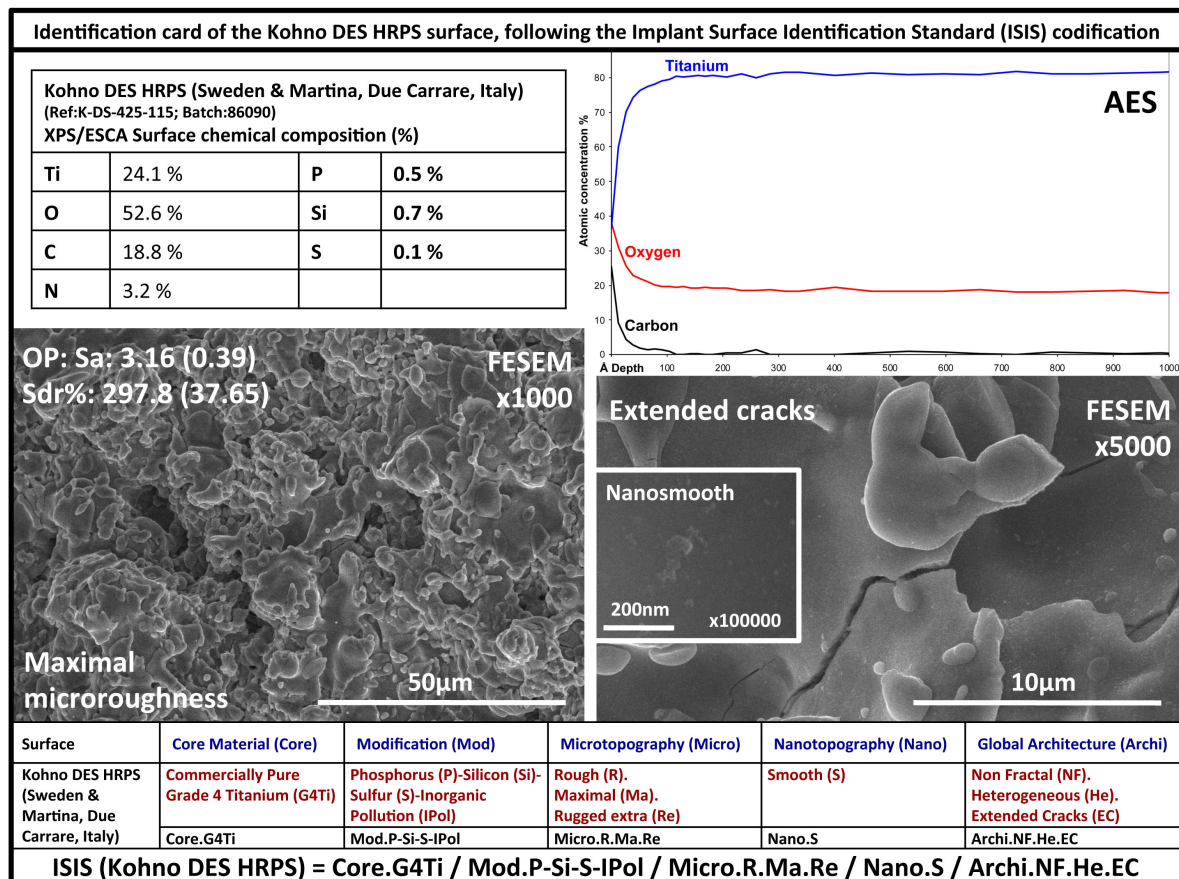


Figure 8. Identification Card of the Kohno DES HRPS surface.

4. Discussion

The concept of surface anodization was developed with the idea of promoting biochemical and biomechanical interlocking of the implant surface with the bone [11]. The anodization process is always producing a very thick micrometric external layer of TiO_2 all over the implant surface, associated with the in-depth impregnation of specific ions (mostly calcium, phosphorus and magnesium at this time), that gives very characteristic patterns of in-depth profile during the AES analysis [2,9]. This characteristic is supposed to promote the chemical interlocking with bone, through an improvement of titanium oxide biocompatibility and bone mineral nucleation [10]. The anodization also creates significant morphological patterns, mostly micropores all over the surface, developed to improve the biomechanical interlocking of the surface. On this matter, companies have developed specific strategies of anodization. Nobel TiUnite was designed to promote a strong biomechanical interlocking, and therefore presented very large micropores and a more aggressive microporosity, associated with very visible extended cracks of the TiO_2 layer. On the contrary, Ospol and Shinhung M were designed with a very smooth microporosity, in order to promote mostly a biochemical interlocking and to avoid the risks of extended cracks (they have only limited local cracks). Other implants (Tecom REP and Cowellmedi INNO) developed intermediate profiles between these 2 extremes, in order to get the benefits of the biochemical and biomechanical interlocking without their respective disadvantages. It is however impossible to determine which strategy is the best at this time [14]. When considering the general evolution of the market, this type of surfaces was a big fashion a few years ago, many companies trying to copy the leader implant company of that time (Nobel Biocare), but this type of surface is nowadays less and less frequent. Even if the literature does not back up clearly and accurately the reason of this slow abandon, the feedback of experience mostly associate this type of surfaces with lower clinical results than other classical surfaces (particularly associated peri-implantitis or simply peri-implant bone loss)[15].

Another approach of light anodization was suggested recently. In the BioSpark surface, the anodization was not tailored to produce micropores, but only to add a final nanopatterning (with nanostructures shaped like nets) as main morphological pattern. Like other forms of anodization, the process is creating a thick micrometric TiO_2 layer with a significant in-depth ionic impregnation (Calcium Phosphate in this case), as observed on the AES profiles of all anodized surfaces. This approach follows the classical concept of biochemical interlocking, but the clinical results of this kind of anodized surfaces remain relatively unknown and not clearly documented.

The concept of TPS surfaces was developed mostly with the idea of increasing dramatically the surface microroughness to have a maximal bone/implant biomechanical anchorage [13]. The spraying of titanium on the surface and its brutal cooling create very typical aggressive patterns on the surfaces and systematic extended cracks all over the external surface layer. This type of surfaces was a big fashion in the early times of modern implantology and is quite rare nowadays [4]. The literature does not back up completely and accurately the reason of this abandon, but this kind of surfaces is often considered to be excessively rough, with the risk of peri-implantitis or peri-implant bone loss [15], and therefore to present lower clinical results than other classical surfaces. Because of this risk, some companies (like the Kohno DES evaluated here) proposed to use this TPS surface in the lower part of the implant and to use a less aggressive surface in the upper and collar part.

However, this last strategy to combine TPS was not significantly followed when considering the global market.

Finally, most surfaces of this group were nanosmooth. Indeed, apart from some rare cases where anodization is used specifically to create some nanopatterns (such as BioSpark or experimental surfaces with nanotubes)[12], microscale anodization and TPS technologies do not produce significant nanofeatures during the deep alteration of the titanium core material. However the development of nanostructures is nowadays often advocated as an important step to improve implant surface performances [16]. This technical limitation of the metallurgy modification processes may participate to the slow abandon of these methods.

5. Conclusion

The surfaces of the first group are mostly the anodized and titanium plasma sprayed surfaces. Each category has specific patterns at the microscale (micropores for anodization, maximal microroughness for TPS), but most surfaces of this group present various kinds of cracks and are in general smooth at the nanoscale, both characteristics being probably related to the concept of alteration of the titanium core material. Only one technology using a final anodization was showing different characteristics. It was also noticed that these technologies are less frequent nowadays, apparently due to the relatively mixed clinical feedback of experience. This explains why only 8 versions of this kind of surfaces were found during the sample collection of this study. Evolutions of these technologies may reappear in the future, particularly as a combination with other styles of surfaces.

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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