Special Review: Consensus Conference

Guidelines for the publication of articles related to implant surfaces and design from the POSEIDO: a standard for surface characterization

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Abstract

Dental implant surface engineering is a very active field of research, however the abundant literature on the topic is often difficult to sort and interpret. Indeed there is a significant lack of homogeneity in the methods to describe the various surfaces available on the market or tested in experimental studies, resulting in confusions in the literature and difficulties to compare the numerous published results. In this article, the POSEIDO (Periodontology, Oral Surgery, Esthetic & Implant Dentistry Organization) is developing and promoting a validated concept for the characterization and description of the implant surface characteristics. The objective of these guidelines is to help researchers to standardize their studies and to promote clarity in this field of research. Illustrated by the description of 2 types of implant surfaces (TiUnite, Nobel Biocare, Gothenburg, Sweden, and Ossean, Intra-Lock, Boca-Raton, FL, USA), these guidelines describe some standardized tools of analysis and terminology that can be used to characterize and define a dental implant surface, particularly its chemical composition (core material, such as titanium, and chemical or biochemical modification through impregnation or coating) and its topography at the micro- and nanoscale (such as microroughness, microporosity, nanoroughness, nanotubes, nanoparticles, nanopatterning and fractal architecture). These POSEIDO guidelines are an important step for the clarification of knowledge and standardization of experiments in this field.

Keywords. Dental implants, osseointegration, titanium.

1. Introduction

The development of implantable materials is an important field of research in medicine in general, and in dentistry in particular **[1]**. Dentals implants are mostly defined by their macrodesign (which is a significant parameter in the clinical indications of the implants)**[2]**, by their mechanical parts (prosthetic components and their accuracy)**[3]** and by their surface **[4]**. Implant design (macroscale) and surface (micro- and nanoscale) of the implants are 2 interconnected parameters that define the interactions of the implanted material with the host tissue, and therefore these characteristics must be well investigated **[4]**.

The literature about dental implant surfaces is currently abundant **[5]**. Many teams and companies around the world are making financial investments to study this topic **[6]**. However, in fact there is very little defined knowledge about what should be an « ideal surface ». The literature is controversial and the published results are difficult to sort and interpret **[1,5]**. The presence of conflicts of interests between researchers and companies may help explain a portion of these problems. However, the true reason of this lack of clarity and consensus is probably more simple: the absence of a relevant standard for the characterization of the studied surfaces **[1]**. In short, researchers are testing many surfaces in vitro (with cells)**[7]** and in vivo (in patients or animals)**[5,8,9]**, but very often they do not accurately describe the surface they are testing. When examining the articles published in the international literature during the last 20 years **[5]**, we can see that researchers often describe their surface by the method of production (sand-blasted acid-etched, blasting with resorbable blasting media, anodization, etc.)**[10]** and not by the detailed characteristics of the surface **[1]**.

For this reason, the POSEIDO (Periodontology, Oral Surgery, Esthetic & Implant Dentistry Organization) intended to define a simple standard to use in surface science and associated publications, so that these works can constitute a more reliable and valuable database for the scientific community. Additionally, this would make these research works easier to understand by the clinician readership **[4]**. This need for well defined classification and terminology exists in all fields **[11,12]**, but it is particularly obvious in surface science. The first step of this strategy was published in 2010 as a general classification and codification system **[1]**. This initiative was followed in 2011 with the publication of the Identification Cards of 14 implant surfaces available on the market **[4,13]**, where these surfaces were fully characterized following the complete codification system previously described.

2. Chemistry and topography, the key parameters

Two levels of characterization can be defined for a dental implant surface **[1,4]**: chemistry and morphology/topography. Both are deeply interconnected and define together the biological properties of a surface **[14-17]**, but they have to be analyzed separately.

The first level is based on the chemical composition of the surface, i.e. the composition of the core material (commercially pure titanium grade 2 or 4, titanium-aluminiun-vanadium alliage Ti6Al4V i.e. grade 5 titanium, zirconia, hydroxyapatite, etc.)**[1,18]** and its eventual chemical (or sometimes biochemical)**[19]** modifications (for example a fluoride or a Calcium Phosphate CaP low impregnation)**[20,21]**. As shown previously, this chemical modification can often be an inorganic or an organic pollution **[4]**. The chemical composition and architecture is a key parameter for the biochemical interlocking between the implant surface and the bone tissue **[1,22-24]**.

The second level is based on the surface topographical characteristics, i.e. the general morphology and structures at the microscale (microrough, microporous, microparticles, presence of cracks or large particles) and at the nanometer scale (nanosmooth, nanorough, nanopatterned, nanoparticled)**[25]**. Several morphological parameters (height deviation amplitude Sa, developed area ratio Sdr%) can be used to quantify this morphology on the microscale **[1]**. The microtopography is a key parameter for the biomechanical interlocking between the implant surface and the bone tissue **[1]**.

The investigation of the nanostructures on the implant surfaces is a recent approach, with potential applications in bone tissue engineering **[25,26]**. The production of surface features at the nanoscale is a new method to control the cell-surface interactions **[27-30]**.

The definition of each characteristic can sometimes be sensitive, and for this reason a classification system and terminology was suggested **[1]**. In the articles about the codification and classification of implant surfaces **[1,4]**, a detailed protocol of characterization was proposed and can be considered as a relevant basic standard. However, many different protocols and instruments exist and allow to gather similar informations.

3. Many techniques of analysis, one objective

Most relevant surface parameters can be characterized using standard analytical tools. We illustrate here these characteristics and analyses with two different commercially available implant surfaces: TiUnite (Nobel Biocare, Göteborg, Sweden)**[10]** and Ossean (Intra-Lock, Boca-Raton, Florida)**[21,31]**.

For the evaluation of the surface chemistry, the use of X-ray Photoelectron Spectroscopy (XPS), also called Electron Spectroscopy for Chemical Analysis (ESCA), can be considered as a gold standard **[32,33]**. XPS is used to determine accurately the quantitative mean atomic composition (in %) and chemistry of a wide and thin surface area (typically 300μm in diameter, less than 20 nm in depth)**[1]**. XPS provides the chemical state of the detected elements, such as the binding forms of phosphorus in phosphates (**Figure 1**). The data provided by this technique may be difficult to understand for a non-physicist, but it is in fact very simple to summarize them in a table with percentages of atomic composition **[4]**.

Auger Electron Spectroscopy (AES) is less accurate than XPS, but it can analyze very small areas and is ideal for checking surface chemical homogeneity, using several repetitive analyses. AES can perform a quick and accurate in-depth chemical profiling of the surface (**Figure 2**)**[32]**. It is thus particularly useful to characterize a core material **[4]**.

A complementary technique called Energy Dispersive X-ray Spectroscopy (EDX) is a simple elemental analysis coupled with the Scanning Electron Microscope (SEM) and allows the identification of particles or structures observed with the SEM (**Figure 3**). The reality is that a wide range of tools can be used to perform the chemical analysis of a surface, for example Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS), Raman Spectroscopy, or even Transmission Electron Microscopy (TEM) after Focused Ion Beam (FIB) cross sectioning of a sample **[34]**. However, most of these techniques require a high degree of calibration to get relevant quantitative data, and do not truly fit to the requirements of osseointegrated surface standardized evaluation.

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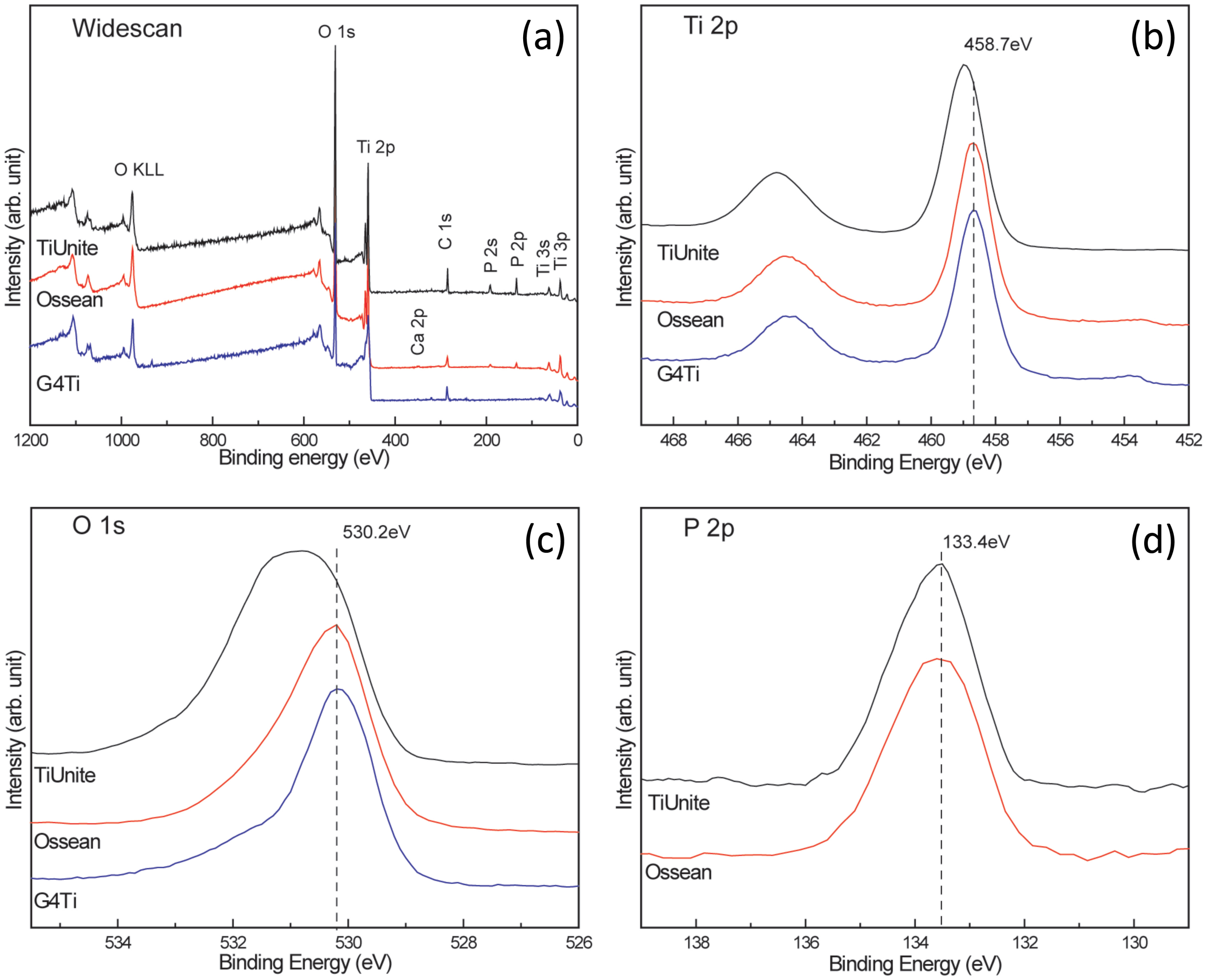

(e)	Ti	O	C	N	P	Ca
TiUnite	17.5	55.1	17	0.9	9.5	-
Ossean	19.8	57	18.6	1.5	2.8	0.3

Figure 1. XPS data of TiUnite, Ossean and G4Ti (grade 4 titanium) surfaces: **(a)** survey XPS spectra; **(b)** high resolution Ti 2p spectra; **(c)** high resolution O 1s spectra; **(d)** high resolution P 2p spectra. Survey XPS data showed major peaks of O 1s, Ti 2p and C 1s for all the samples and minor peaks of P 2p for TiUnite and Ossean. In P 2p high resolution spectra, there was no significant difference in peak position and spectra shape between TiUnite and Ossean. On the contrary, Ti 2p and O 1s spectra of TiUnite showed higher peak positions and wider peak shape than the spectra of G4Ti and Ossean. TiUnite is indeed an anodized surface, with phosphorus high impregnation within a micrometer thick titanium oxide TiO_2 layer, and with thus formation of titanium phosphates. On the other hand, Ossean shows a calcium phosphate low impregnation that negligibly altered the surface chemistry of TiO_2. The results of these XPS analyses are also reported in a more simple and reader-friendly way as percentages of atomic composition for each element **(e)**.

(O as oxygen, Ti as titanium, C as carbon, N as nitrogen, Ca as calcium, P as phosphorus)

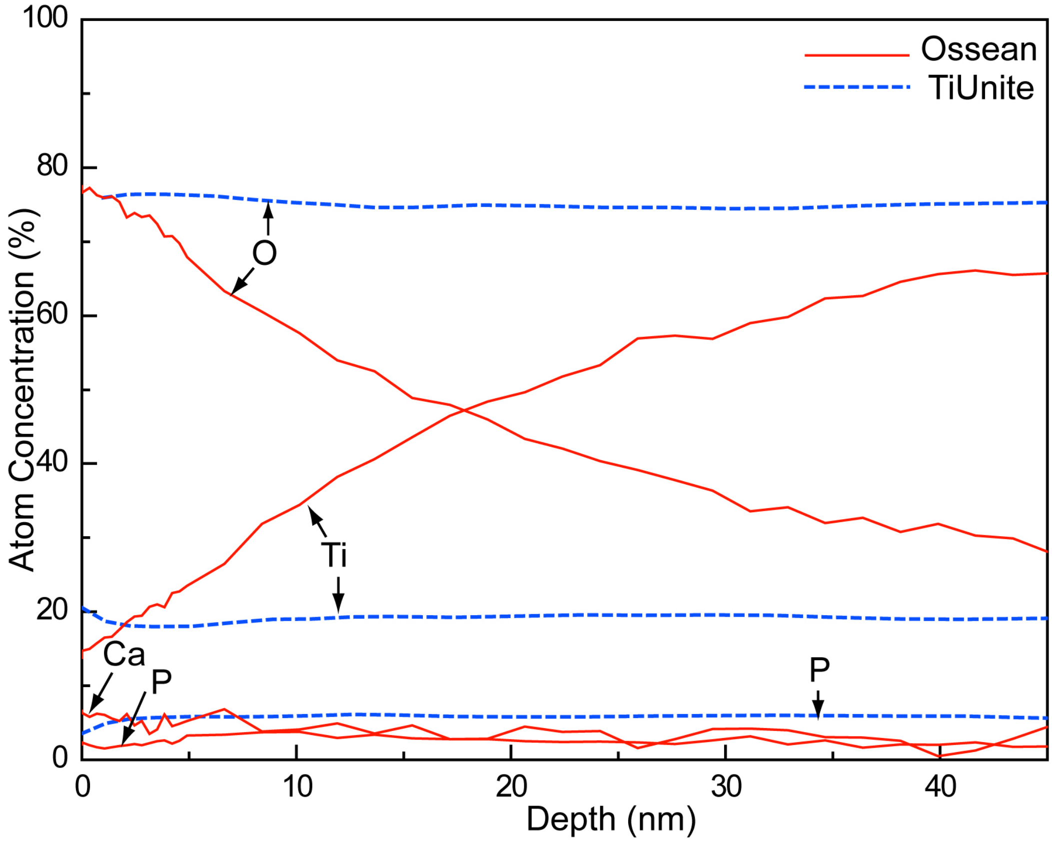

Figure 2. AES in-depth profiling of TiUnite and Ossean surfaces down to 45nm. The two surfaces show completely different patterns. TiUnite is anodized and thus presents a thick and homogeneous TiO_2 layer highly impregnated with phosphorus. Ossean is based on another technology, with a decreasing proportion of TiO_2 and a stable CaP low impregnation along the in-depth profile of the surface. (O as oxygen, Ti as titanium, C as carbon, Ca as calcium, P as phosphorus)

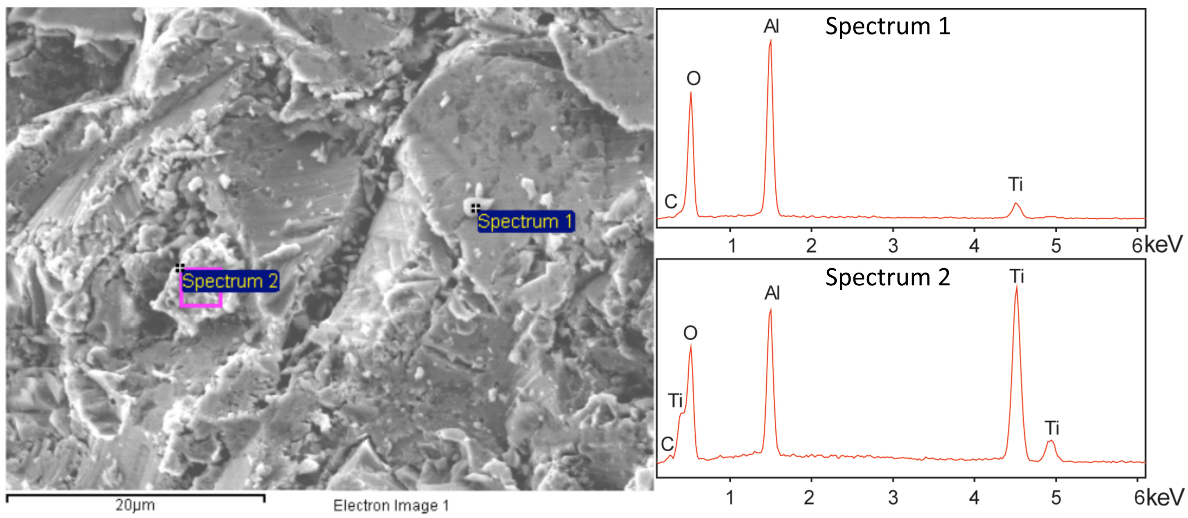

Figure 3. Composition analysis with EDX probe of particles observed with SEM. The surface of this early version of this Ankylos implant (Friadent, Mannheim, Germany) is covered with microparticles. The EDX analysis allows to identify these particles as Aluminium Oxide blasting residues. Spectrum 1 was acquired in a very small area, showing clear Al and O signals. Spectrum 2 was acquired using a larger interaction volume, resulting in clear signals of both the AlO residual particles and the Titanium oxide below.

The topography can be assessed with many different tools, but two are particularly adapted and common. Scanning Electron Microscopy (SEM) is the gold standard for morphology characterization at the micrometer level (SEM with tungsten source)**[32]**. However, Field Emission-SEM (FE-SEM) is required to increase the analytical resolution, and to observe and characterize the nanotopography and associated nanostructures (**Figure 4**)**[4]**. Without FE-SEM, the analysis of the nanostructures should be considered as incomplete and inadequate, even if the authors may have the feeling to observe something relevant **[1]**. This is a problem of resolution, and using the wrong instruments simply creates artefacts.

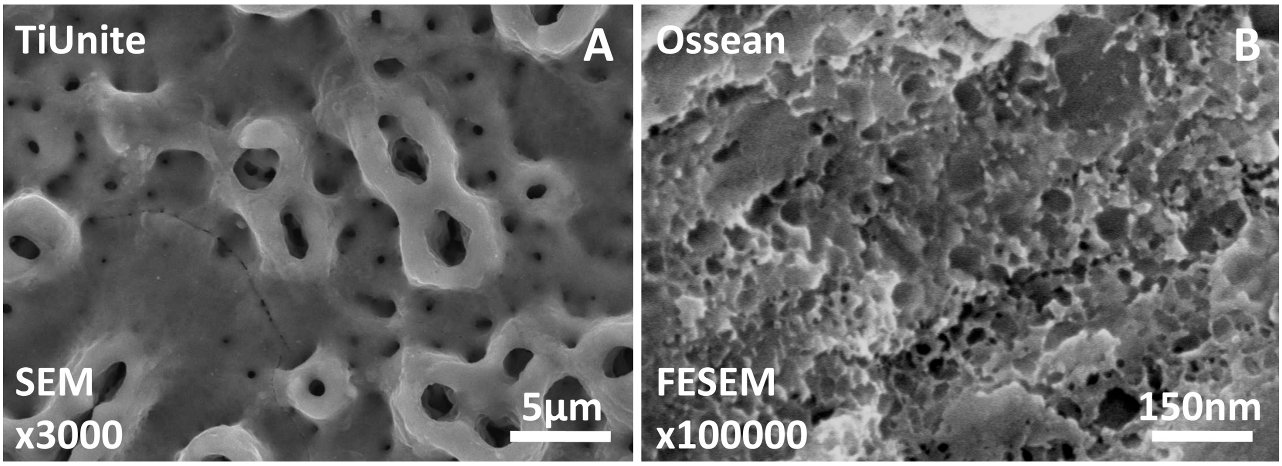

Figure 4. Scanning Electron Microscopy (SEM) examination. A. TiUnite is an anodized surface with a typical microporous topography and cracks observable with the classical SEM at low magnification. **B.** Ossean is a microrough surface presenting a typical nanoroughness observed at higher magnification. However without the use of a Field Emission-Scanning Electron Microscope (FE-SEM), it would be impossible to observe so clearly the nanostructures, particularly in this environment rich in CaP.

Interferometer (IFM) or optical profilometry (OP) is an efficient tool for the evaluation of the microtopography general aspect and quantitative parameters on wide areas (**Figure 5A**)**[4,8]**. A FE-SEM can also be coupled with a metrology software to produce 3D reconstructions of the surface (stereo SEM) and to perform a quantitative morphology analysis, both at the micrometer and nanometer level (**Figure 5B**).

All these techniques have their advantages and limitations. This list of instruments is not exhaustive, and all these analyses are not required to publish an article about surfaces. However, it should be now mandatory for the authors to provide a clear and detailed chemical and topographical characterization of the tested surfaces if they want to have their article considered for further review in an international journal. The POSEIDO suggested characterization system offers a strong coherence and an easy way to clarity, even if all protocols offering similar information are acceptable. This endeavour is an important step for the development of a high quality database about dental implant surfaces, and also to simplify the understanding of basic science surface articles by the clinician readership.

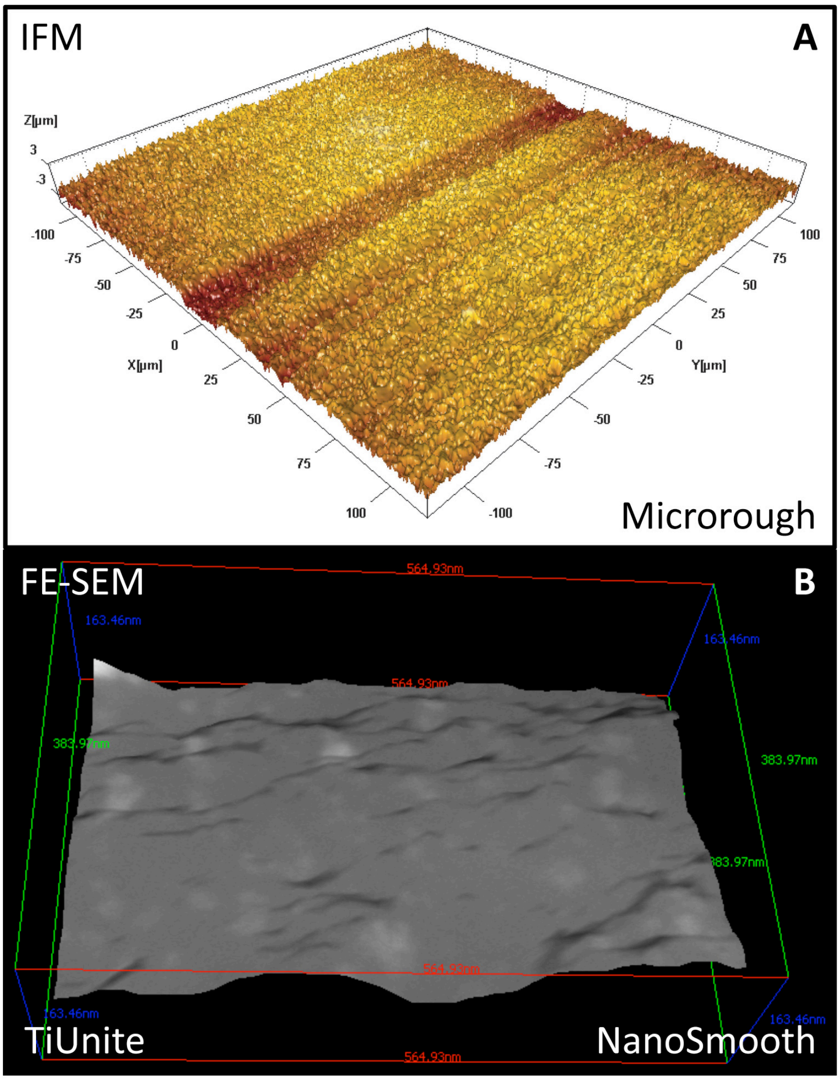

Figure 5. IFM and computed FE-SEM evaluation of implant surfaces. A. IFM is an easy and powerful tool for quantitative evaluation of the microroughness values on wide surface areas (typically 230µm x 230µm). **B.** FE-SEM analysis coupled with a metrology software allows to perform a quantitative morphology down to the nanoscale. This TiUnite nanometric square surface shows an almost flat nanotopography, and is considered as nanosmooth.

This protocol for surface has also to be considered for all articles about implant macrodesign. Indeed, testing a new design always implies to rule out the potential bias related to surface. The first step is therefore to characterize carefully the surfaces, to be sure that they are strictly the same between the samples, before proceeding further for the new design testing. In the literature, the surfaces are rarely checked before testing different designs, and this may explain why the published results in the international literature are so difficult to sort and interpret.

4. Conclusion and Perspectives

This consensus article is a first step of the POSEIDO initiative to develop common standards in the field of implantable biomaterials. These general guidelines for surface characterization offer a simple standard method for the research in this field, to improve the

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quality of the experiments and to clarify the literature. When more results will be published using this approach, it will be possible to sort and interpret more easily the data on this topic, and to refine our knowledge. These general guidelines are a first important instrument, and should be completed in the future with the feedback of experience.

Disclosure of interests

The authors have no conflict of interest to report.

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