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Numerical characterisation of biomedical titanium surface texture using novel feature parameters

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ABSTRACT

In biomedicine, titanium materials with specific surface textures (for example, those produced using consecutive polishing, sandblasting and acid etching) are widely used to facilitate osseointegration of human/animal tissue with implant surface structures. The surface texture has a critical role on the material's functionality in terms of cellular adhesion and proliferation. However, conventional surface topography characterisation parameters pertinent to cellular attachment such as *Sds* (density of summits of a surface) and *Ssc* (arithmetic mean summit curvature of a surface) are liable to be influenced by low amplitude high spatial frequency components or measurement noise. In this research, a novel feature characterisation, based on pattern recognition techniques is implemented on specially processed titanium surfaces. The mean dimensions and densities of the micro-scale features of the surfaces are extracted. The statistical analysis results demonstrate the efficiency and stability of the feature analysis compared to the use of conventional surface texture parameters. Additionally, potentially efficient parameters for characterisation of biomedical surfaces are indicated through the use of a one-way analysis of variance. As a case study, from the point of view of surface metrology, limited experimental data is presented; the intention of the authors is to give a guide to innovative use of the novel surface characterisation techniques. A large amount of biomedical experiments would be needed in the future to fully validate the correlation between the surface texture parameters and its biomedical functions but the present work provides a useful start point for a larger study.

1. Introduction

Surface topography analysis plays an important role in the functional prediction of properties such as friction, wear, lubrication and adhesion for engineered surfaces. In biomedicine, roughened titanium surfaces have also proved beneficial to the adhesion and growth of human or animal osteoblast cells [1,2], leading to rapid biological fixation for implants. Typically the roughened titanium surfaces which promote rapid osseointegration are fabricated by consecutive polishing, sandblasting and acid etching [1]. The surface topography produced by such processes is multi-scale in nature and consists of large scale (hundreds of micrometers) and micro-scale (several micrometers) surface features. Significantly, these micro-scale geometric features influence osteoblast proliferation and differentiation in cell culture. For example, Hatano's results [3] show that cellular proliferation levels increase in response to the increase in the degree of surface roughness up to 0.8 μm (*Ra*) and then decrease to the level observed for the smooth surface. The conclusions of Mustafa [4] indicate that the proliferation and differentiation of cells can be enhanced by increasing the particle size up to 300 μm used in the blasting process. However, the accurate role of surface topography on osseointegration remains poorly understood.

Conventional surface texture characterisation parameters such as *Ra*, *Rt* and *Rz* [3] of a surface profile or *Sa*, *Sds* and *Sdr* [4,5] on an areal surface have been used to ascertain the functionality of titanium surfaces and have been proven to be of limited use. However, surfaces are complex entities which cannot be described completely by a single or even a few numerical descriptors [6]. Numerous numerical parameters have been proposed in the past most of which are now redundant [7]. For example, *Sa* and *Sq* are a pair of highly correlated parameters. Only one of them should be selected for use in most applications. Choosing a set of significant parameters which are functionally correlated is a notoriously difficult topic. The solutions in engineering are usually based on a large amount of empirical experimental data [8]. Very little progress has been made in this area except where an excellent correlation

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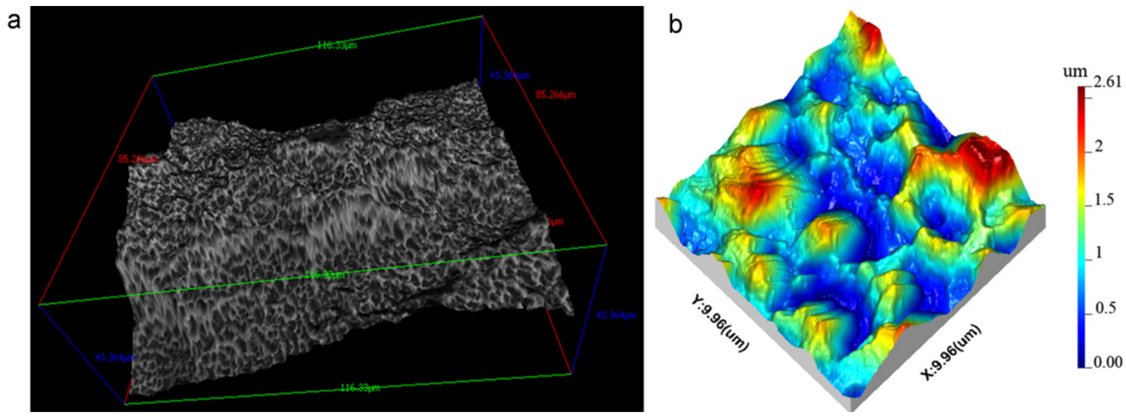

Fig. 1. (a) Stereo SEM image of a representative titanium sample surface. (b) AFM image of sample S3 (form removal by levelling and 8 μm L-filtering).

model has been developed, by which the functionalities and the surface geometric characteristics are connected. In the recent few years, some generic methods have been suggested or used, such as parameter classification [6,8], Analysis of Variances (ANOVA) and correlation analysis [9]. In this case study, the ANOVA method is adopted as a tool to show functional correlation.

Specifically when considering the surface topography of titanium bio-implants, one of the main elements is the micro-scale concave pore features generated by acid etching, which lay on the relatively large scale sandblasted areas (see Fig. 1a). Clearly, conventional profile (two-dimensional) or even areal (three-dimensional) statistical based parameters are deficient in separating and quantifying these multi-scale geometrical features. It is considered that multi-scale roughness descriptors quantifying the mean spacing between the micro-features, mean depth or their densities would provide improved tools for predicting the surface functionality.

The draft standard ISO/FDIS 25178-2 [10] provides a robust solution for this type characterisation challenge. It not only retains the most typical conventional parameters but also proposes several novel solutions such as pattern recognition techniques. Two parameter sets are defined as "field parameters" and "feature parameters". The field parameter set applies statistics to the continuous point cloud of the areal surface data [11] which contains the majority of the conventional areal surface texture parameters. The "feature parameters" apply statistics to a subset of predefined topographical features [12] such as significant peaks yielding parameters such as *Spd* (density of peaks of a surface), *S5v* (five point pit height of a surface), *Spc* (arithmetic mean peak curvature of a surface). The basic philosophy behind feature parameters is to treat a surface as a collection of features (edge and facet features or Maxwellian features: hills, dales saddle points, ridge lines and course lines) [13]. A surface is segmented into a series of regions that containing individual topographically significant features. The segmented surface geometrical features can then be analyzed individually or statistically. A feature characterisation toolbox – "five steps to a feature parameter" – has been defined [10].

In this research, advanced parameter sets [10] are utilized to characterize the micro-features that are generated mainly by the acid etching process of titanium bio-implant surfaces. The densities and dimensions of the micro-pores generated are calculated innovatively by means of the feature characterisation toolbox. Based on a one-way analysis of variance (ANOVA) [14], the potential significant parameters are indicated. The higher significance of the feature parameters is demonstrated compared to the field parameters. It should be borne in mind that selection of significant parameters that are functional correlated requires a large amount of experiments and empirical data.

As a case study, from the point of view of surface metrology, limited experimental data is presented here; the intention of the authors is to give a guide to innovative use of the novel surface characterisation techniques. A large amount of biomedical experiments would be needed in the future to fully validate the correlation between the surface texture parameters and its biomedical functions but the present work provides a useful start point for a larger study.

2. Measurement conditions

Three representative titanium surface samples processed by the National Physical Laboratory (NPL), which are coded as S1, S2 and S3, were used for this study. The surfaces were fabricated by polishing, sandblasting, followed by acid etching. A stereo scanning electron microscope (SEM) and an atomic force microscope (AFM) were used to measure the three sample surfaces at the micrometer scale. An image captured by the SEM of the etched surface is shown in Fig. 1(a). The numerous micro-pores generated mainly by the acid etching process are clearly resolved. The AFM was then used to measure the geometric form of the micro-cells. Due to the re-entrant and relatively rough nature of the surface features, a sharp tip used in tapping mode (cantilever 910M-NSC15) was implemented to minimise the smoothing effect due to tip geometry. Two repeated measurements were carried out for each sample. A typical AFM measurement result (wavelengths over 8 μm filtered out) is shown in Fig. 1(b).

The protocol used for choosing the sampling conditions was as follows. Firstly, the instrument capability and the size of the features to be measured determine the sampling area. For example, for a particular micro-scale geometric structure, the minimum sampling area should cover at least a representative number of critical features. In order to make full use of the bandwidth and band resolution of the instruments employed, the maximum sampling size was chosen which was 256 by 256 points. To obtain statistically significant data, several continuous features should be covered within a sampling area. If the sampling area is too small it may not contain enough information and have less statistical significance. Thus the sampling area in this work was set as 10 μm × 10 μm (the scale of the micro-features was around 2 μm from visual examination of the SEM images and from previous experience).

3. Field parameters

The field parameters comprise of a number of different groupings: 'height parameters', 'spatial parameters', 'hybrid parameters', 'functional parameters' and others. After form removal by sequen-