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Characterization of nanostructured material images using fractal descriptors

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ABSTRACT

This work presents a methodology to the morphology analysis and characterization of nanostructured material images acquired from FEG-SEM (Field Emission Gun-Scanning Electron Microscopy) technique. The metrics were extracted from the image texture (mathematical surface) by the volumetric fractal descriptors, a methodology based on the Bouligand–Minkowski fractal dimension, which considers the properties of the Minkowski dilation of the surface points. An experiment with galvanostatic anodic titanium oxide samples prepared in oxalyc acid solution using different conditions of applied current, oxalyc acid concentration and solution temperature was performed. The results demonstrate that the approach is capable of characterizing complex morphology characteristics such as those present in the anodic titanium oxide.

1. Introduction

The morphology analysis of solid samples is an important research area in Materials Science to characterize some of their properties [1–4]. Generally, the technique employed in this kind of application consists in the quantitative analysis of the micrographs obtained using one (or more) of the following techniques, such as FEG-SEM (Field Emission Gun-Scanning Electron Microscopy), AFM (Atomic Force Microscopy), STM (Scanning Tunneling Microscopy) or TEM (Transmission Electron Microscopy). In any case, the results are a matrix of values which expresses the topography of the measured sample.

When the morphology of the material is investigated, we observe that each image presents a specific distribution pattern. This distribution is quite similar to that found in textures classically studied with image analysis tools, e.g., the so-called texture analysis methods [5]. Of course, the quantitative analysis of nanostructured materials is easy to perform for well behaved samples which have been described using simple functions of existing software packages. One example is the automatic counting of pores in self-organized anodic porous alumina [1] using ImageJ [6] and Gwyddion [7]. The problem is different when studying complex morphology characteristics of the samples as those ones where the distinction among the patterns is not obvious. In this case, a more sophisticated analysis must be used and these are generally are not included in those software packages described above.

Among the methods described in the literature, those ones based on fractal analysis have presented excellent performance in the investigation of complex textures, mainly on those synthesized and natural samples [8–11]. Actually, nature is rich in self-similar patterns, that is, structures which repeat themselves under different scales. From a mathematical point of view, this is also an intrinsic property of fractal objects. Therefore, fractal geometry is appropriate to measure such kind of structures and, as consequence, self-similarity also measures complexity (meaning the level of details along scales) which is directly related to spatial occupation in the structure.

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Table 1
Experimental matrix for the anodic titanium oxide samples preparation using a factorial design procedure.

Experiments	Current density/mA cm^{-2}	Temperature/°C	Concentration/mol L^{-1}
1	10	10	0.05
2	20	10	0.05
3	10	30	0.05
4	20	30	0.05
5	10	10	0.5
6	20	10	0.5
7	10	30	0.5
8	20	30	0.5

Not withstanding that fractal dimension provides a good solution in many object identification problems, it is limited in the representation of some classes of objects [12] due to two main aspects: (i) fractal dimension is a real number which is insufficient to characterize such objects. (ii) there are samples that have different patterns but present the same fractal dimension. In fact, these objects can have a more or less self-similar aspect depending on the scale observed [13]. In the literature, different propositions have been presented to solve the above drawbacks. The main ones are multifractal [14], multiscale fractal dimension [15] and fractal descriptors [9]. Considering that several papers have demonstrated the superior performance of fractal descriptors dealing with texture images over the other ones, we are focused here on such an approach as a tool for texture discrimination [9,10,5,11].

We apply Volumetric Minkowski descriptors methodology, which was initially developed in Ref. [8]. It is derived from the Bouligand–Minkowski fractal dimension. The descriptors are obtained by mapping the original gray level image (of FEG-SEM data, in this case) onto a three-dimensional mathematical surface. Thus, such a surface is dilated by the Bouligand–Minkowski method using spheres with predefined radii. The fractal descriptors are then estimated from the volume of dilation for each sphere radius. With the growing of the dilation radius, the spheres start to interfere among themselves, forming a wavefront which is tightly related to the structure of the material. It is important to stress that the dilation process captures the arrangement of the topography [8]. Thus, these descriptors are capable of providing very rich information about the morphology of the material and, consequently, are a strong method for a nanostructured material characterization task. The use of the Volumetric Fractal Descriptors applied to nanostructured surfaces was initially proposed in Ref. [16]. In that seminal project, it was suggested that Fractal Volumetric Descriptors could be used to characterize and analyze such nanostructures, showing the discrimination power on two distinct conditions. In the present work, an experiment with galvanostatic anodic titanium oxide samples prepared in oxalyc acid solution using eight different conditions of applied current, oxalyc acid concentration and solution temperature was performed, and demonstrates that the proposed technique is capable of identifying the nanosurfaces. The nature of the material's surfaces and its images are a difficult problem in image analysis, and the proposed technique demonstrates itself to be suitable to characterize and to identify nanostructured surfaces.

2. Materials and methods

2.1. Materials

The samples used in this work were galvanostatic anodic titanium oxide ones prepared in oxalyc acid solution. In this electrochemical preparation method, a titanium plate is the anode in a two electrode electrochemical cell. A platinum plate was used as a cathode. Then, the anode is polarized under constant current condition and an oxide film starts to form over the anode following the equation: $Ti + 2H_2O \rightarrow TiO_2 + 4H^+ + 4e^-$. It is important to stress that TiO_2 is formed by the direct reaction between the metal and water over the metal. The surface morphology of the oxide is sensible to the experimental conditions used. In the present case, different values of applied current, oxalyc acid concentration and solution temperature were used as described in Table 1. Using a 2^3 factorial design [17], 8 titanium oxide anodizations were performed, generating, therefore, 8 classes of samples. From each class, 8 images from different regions on their surface were acquired. Therefore, we have a total of 64 samples to be used in the model building. Each sample is a rectangular piece of the plate, which is measured through SEM-FEG technique generating a matrix (image) with a resolution of 3072×2060 pixels. Fig. 1 shows one image per class, illustrating the general aspect of the dataset.

3. Results

3.1. Fractal theory

Fractals are objects formally defined as a set of points whose Hausdorff–Besicovitch dimension (see the concept below) exceeds strictly the Euclidean dimension. In practice, it is an object generated through a dynamic system that presents infinite complexity and self-similarity [13]. Here, complexity states for the level of details under different scales.