Tube Center Detection in SEM Images of Titania Nanotube Arrays

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***Abstract*—We propose an effective method for detecting the nanotubes centers in SEM images representing Titania Nanotube Arrays (TNAs). Using several image processing steps we are able to detect with fair accuracy the centers of the nanotubes and to estimate a relative large number of nanotubes. This allows physicists to easily assess the quality of their produced TNAs, as manually counting nanotubes is practically impossible.**

Keywords—titania nanotube array; image processing; SEM images

I. INTRODUCTION

There exists a continuous interest in titania coated materials due to their remarkable properties in terms of chemical and thermal stability, mechanical hardness, optical transparency, high refractive index, photocatalytic activity and bactericidal ability [1]. Such properties made them ideal to be used in environment protection, medicine, various industries and energy conversion. A special kind of TiO_2 materials is the titania nanotube array (TNA), a nanostructure prepared, for instance, by electrochemical corrosion of a Ti surface. In specific conditions, the dioxide develops itself as highly ordered nanotubes. Their geometrical characteristics can be controlled by selecting the electrolyte content, discharge current and duration [2-7].

In the present work we use several top-view high-resolution field-enhanced secondary electron microscopy (FE-SEM) images of TNA samples [7]. The preparation has been done via surface anodization in ammonia-based electrolytes.

The images have been acquired on a Cross Beam System (NEON 40EsB) from Carl Zeiss with electron beam resolutions of 1.1 ÷ 2.5 nm and magnification ranging between 12× and 2,600,000×, equipped with EDS, SE, in-lens and BSE detectors. The details of the specimen surface and a good topographic contrast have been achieved by using a low enough voltage of 5 kV.

Since such TNA images can contain tens of thousands nanotubes (see Fig. 1), even the manual quantitative assessment of their number is almost impossible or its accuracy can be questioned. A solution to fulfill this task is to use image processing and pattern recognition techniques [7-9] in order to automatically detect the position of nanotubes inside an image, followed by automatically counting them and computing of parameters like the percent of aria covered by oxide, the percent of aria covered by nanotubes, the average diameter of nanotubes and the average density of nanotubes per square micron.

In [7] the authors used the watershed transform as an evaluation method for TNA structures, with good results. Automatic counting has been successfully performed, but with some imitations due to incorrectly detected contours in the difficult shaded regions. A manual method to compare with is unpractical in evaluating *nanostructures* due to the very high number of tubes. Interesting results have been also obtained by using a technique based on texture analysis and support vector machines classification, described in this paper.

In [9], the same watershed transform has been used to isolate titanium alloy individual *microstructural* features, such as the grains and alpha lath colonies. Thus, the obtained results have been good and the time to process a fully lamellar microstructure has been decreased from the manual evaluation in 15 minutes to less than 15 seconds for the automatic procedure.

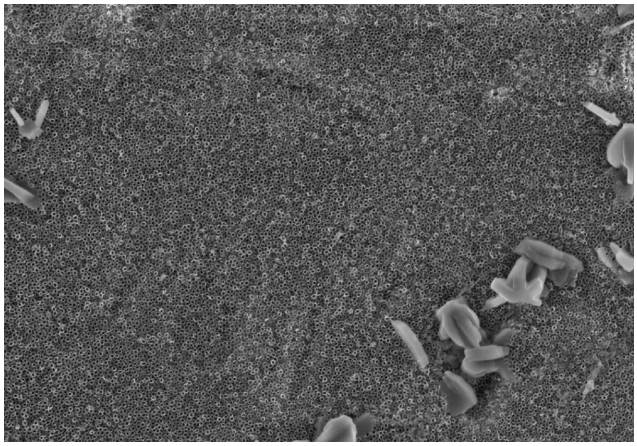

Fig. 1. An original SEM image (1024×711 pixels) with several thousand nanotubes

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II. THE ESSENTIAL STRUCTURE OF A NANOTUBE IN A SEM IMAGE

Basically, a nanotube has an inside empty part (the hole), consisting of dark pixels (black or almost black), and a surrounding ring of much brighter pixels (see Fig. 2, right). The ring can touch other rings of neighbor nanotubes, some holes between nanotubes or some extended oxide areas.

Our proposed method for detecting nanotubes existence and position is a structural one, based on detecting first the darker pixels in the gray level SEM image (the pixels with a value under a determined threshold) and consider them as a start for detecting all the components of a nanotube. Applying the threshold results in the formation of small islands of dark pixels, for which we can compute the centroid pixel. Then we can search around this pixel for the darkest pixel and consider it as the center of the nanotube hole.

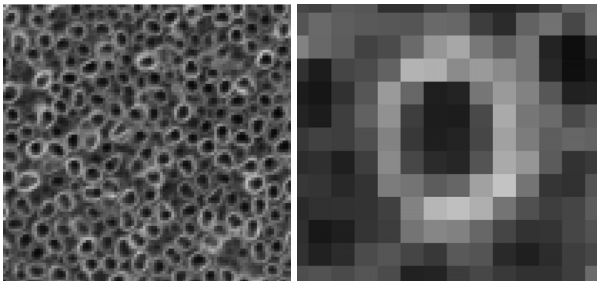

Fig. 2. Detail of the image in Fig.1, showing a relative homogenous area of nanotubes (of about one square micron - 101×98 pixels) and an enlarged area representing one nanotube (each pixel being represented by a small square)

III. IMAGE PROCESSING STEPS AND RESULTS

In order to test this method, we have implemented it in a MATLAB script. The values of some parameters have been established empirically, working on small parts of whole SEM images (see Fig. 2, left). Thus it was easier to assess the result of detecting the centers of nanotubes and we have changed the parameters until we obtained almost the maximum number of detected nanotubes on such small image parts.

The first step is to dissociate the pixels in the image that pertain to oxide and holes. We have found that a threshold of 65 makes us sure that all pixels brighter than it are not pertaining to nanotube holes and could be considered as part of the oxide. Consequently, the same threshold of 65 assures a pixel darker than it is either part of a hole of a nanotube or of the dark regions between nanotubes (see Fig. 3). This particular threshold is extremely important and the number of detected nanotubes depends very much on it. We have tested our method on several SEM images and in a few cases it was necessary to change this threshold in order to obtain credible results.

In the second step, we have found all the islands of connected red pixels and selected from them only the ones that consists of more than 1 pixel and less than 21 pixels. These kind of selected islands have their dimension in pixels appropriate to represent nanotube holes.

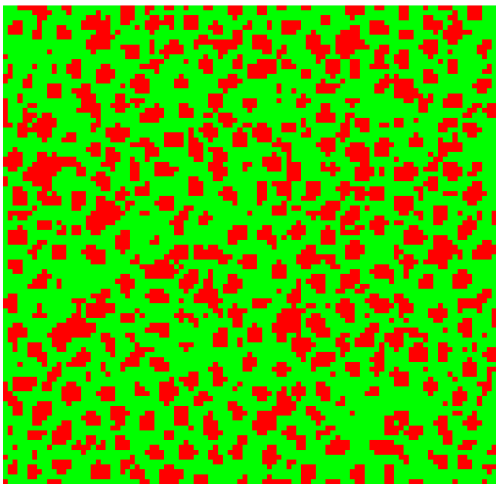

Fig. 3. Combined image of pixels marked as representing oxide (in green) and holes (in red), obtained by thresholding the detail image in Fig. 2, left

Then we extract the centroid of each selected red island and obtain a first candidate for a nanotube center, like in Fig. 4.

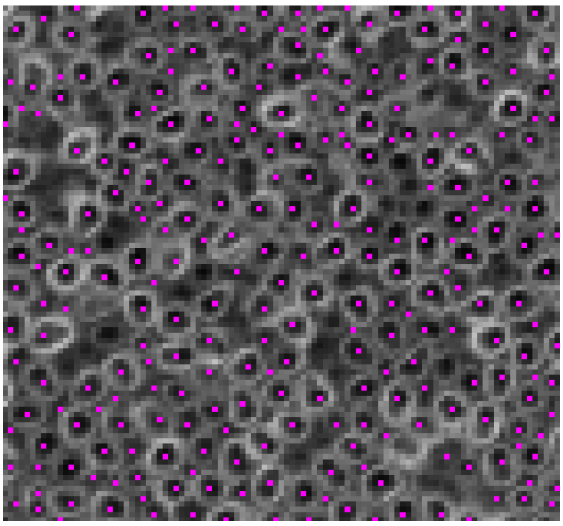

Fig. 4. The first candidates of nanotube centers obtained from the thresholded image in Fig. 3 and superimposed on the initial detail image

In the last step, each center candidate has been taken into consideration and a search has been done around it for darker pixels. If such a pixel is found, then this pixel will become the final nanotube center. Also, a search has been done for nanotube centers situated too close (less than 2 pixels).

In such a case only one nanotube center is preserved (the darker one), the other one being deleted. Fig. 5 presents the final detected nanotube centers.

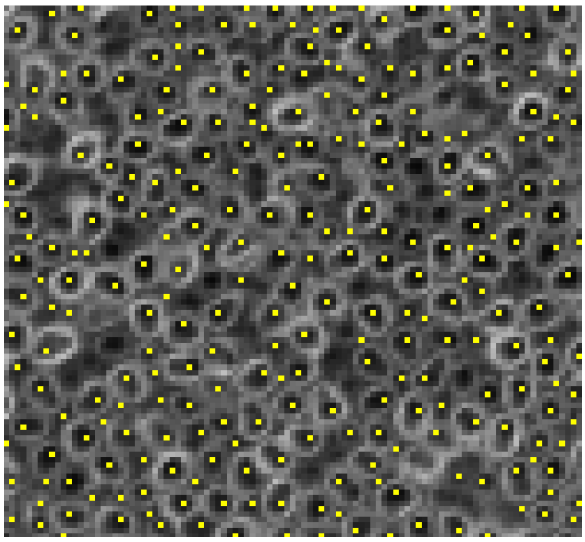

Fig. 5. The final detected nanotube centers

Our method has detected 287 nanotubes in this small area of approximatively one square micrometer (101×98 pixels). In Fig. 6 we can see in red the first ring of dark pixels (with their intensity values exceeding at most 20 gray levels) around the nanotube centers.

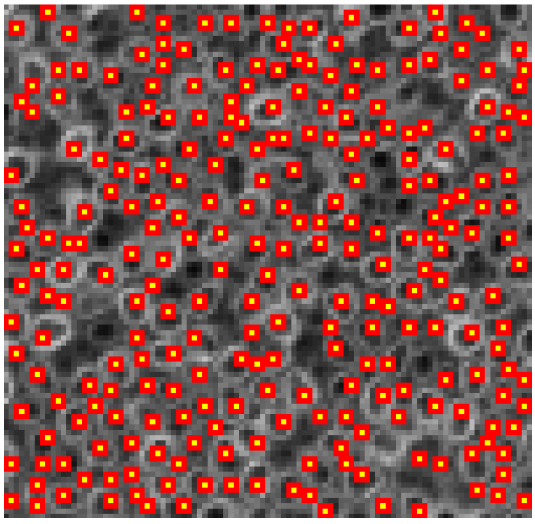

Fig. 6. The final detected nanotubes

Applying the proposed method to the whole SEM image presented in Fig. 1 results in the nanotube detection that can be seen in Fig. 7. There are 18.960 detected nanotubes, a very large figure, making the manual counting practically impossible.

In our tests we have used a series of 12 whole SEM images. From each of them a representative small part has been taken in order to manually assess the effectiveness of our method. Also the parameters have been adjusted so that the results are consistent for all of the 12 SEM images. We have detected from 12.000 to 25.000 nanotubes on each image and there have been two images where the results were not consistent, either because of their different content or because there has been a significant difference in the overall brightness.

At this stage of our research it has not been possible to manually count the nanotubes present on a whole SEM image. However, when adjusting the method parameters on small areas, like in Fig. 5, we have found that the missed neurons have been almost compensated by the falsely detected neurons, therefore the estimated number can be considered a good one. Later stages will also address the possibility to identify the initially missing neurons and to discard the falsely detected ones.

IV. Conclusions and Further Research

We have proposed an effective and fast technique for detecting the nanotubes centers in a SEM image representing a Titania Nanotube Array. Using several image processing steps we have been able to detect with sufficient accuracy the centers of the nanotubes and to estimate the number of nanotubes on a relatively large image.

Observing the results, most of the nanotubes have been detected correctly. There are a significant number of nanotubes that can still be observed, but are not detected, due to the fact that these nanotubes are either shadowed by the surrounding ones (probably much taller) or their gray level composition is not the same as the majority.

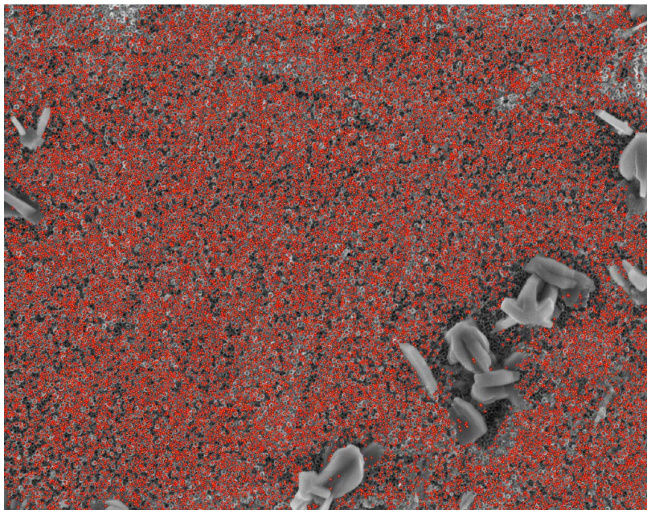

Fig. 7. The final detected nanotubes for a whole SEM image

We intend to develop special routines to try to detect even these nanotubes, normally situated inside large areas of contiguous dark pixels. Running the same detection process

only on these areas, but with appropriately altered parameters, can increase the rate of nanotube detection.

Finally, there are some nanotubes detected in the oxide areas, where accidentally some pixels are a little darker. At this point of our research these nanotubes cannot be discarded. However, in the next steps of finding all the pixels belonging to the nanotube holes and to the surrounding ring, these nanotubes will be invalidated since they will have a small computed diameter.

We intend to continue our research with detecting each part of a nanotube, thus giving us the possibility to compute an average diameter for the nanotubes of a SEM image, the density of nanotubes and the percentage of a SEM image occupied by nanotubes and by the oxide. These are important parameters to assess the success and quality of the growing TNAs.

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