Image processing. Application to the characterization of thin films

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Abstract. Image processing and digital image acquisition became on last decades most valuable tools in the characterization of materials. The strong development of micro and nano sciences and technologies on recent years brought special demands to the non-invasive inspection and characterisation of thin films and nanostructures. Digital image processing can be successfully applied to different types of microscopy' images but also together with other characterization methods like microtopography spectroscopy or lifetime measurements. On this communication we will report on the work developed on the field at University of Minho at the Department of Physics' Microtopography Laboratory with samples provided by the department' Functional Coatings Group.

1. Introduction

Low cost high computational power microcomputers and digital cameras, image digitizers and processing boards allowed a major development of Image Processing (IP) and its applications[1-7]. It is also being registered the development of an enlarged set of IP methods and techniques. Most phenomena and processes in the physical world can be represented by bi- or multidimensional functions that with this development in IP can be suitably analyzed understood and processed.

According to the even more conservative estimates more than 75 percent of all information received by the man is visual. Furthermore its processing efficiency is remarkably high. In this context an enlarged set of efficient hardware software and IP methods and tools is now days readily available for application in the widest variety of fields in Science and Technology. In what concerns non-invasive inspection and characterization of micro and nano structures this is definitely also true.

Typically image processing (IP) deals with images which are two-dimensional entities (such as digital images, scanned photos or graphs, x-ray films, microscope images, satellite pictures, …) captured electronically using a scanner or camera system that digitizes the spatially continuous co-ordinates resulting in a regularly spaced matrix filled with values within a discreet range.

A digital image is a mapping from the real three-dimensional world to a set of two-dimensional discrete points. Each of these spatially distinct points holds a number that denotes intensity, amplitude, grey level or colour for it, and can be conveniently fed to a digital computer for processing. Here, processing essentially means algorithmic recognition or analysis, enhancement and manipulation of the digital image data. Every image processing technique or algorithm takes an input, an image or a sequence of images, and produces an output: a modified image or a set of parameters and functions describing the content of the input image [1-14].

Although digital image processing is, by far, the most important one, analogue optical IP, including in real time, have also a major role. For instance the implementation of Fourier processing techniques in the frequency domain is given excellent results especially in the study of dynamic or transient processes. Furthermore the concept of image as bi-dimensional entity can be easily generalised to 3D or even multidimensional image like structures processed with essentially the same tools as the conventional 2D images.

The interest of the application of IP in surface science seems obvious. Dependent on the scale, direct or indirect visual evaluation of the surface' structure is always sought. In micro and nano science and technology this is also true. A large number of characterization tools and methods produces as output two dimensional structures that can be seen as images and treated as such.

In fact Image Processing can be applied at smaller or larger extent to virtually all the range of available NDE techniques [14] including:

- optical interferometer and profilometers or microtopographers (ie. Nomarski and confocal microscopes, depth of focus, speckle, structured lightning, triangulation, …),
- difractometers (Angle-Resolved Scattering, Total Integrated Scattering),
- elipsometers,
- spectroscopes and fluorescence spectrophotometers,
- photoacoustics and life-time spectroscopy systems,
- SEM,
- STM,
- AFM (in Figure 1 is presented an AFM micrograph of a thin film),
- Raman spectroscopy,
- XRD (in Figure 2 a processed XRD image of a μc-Si:H film is presented [15]),
- FTIR,
- Eddy currents,
- induced magnetic fields,
- thermal waves analysis,
- ultrasonic waves,
- X-ray,
- shearographers,
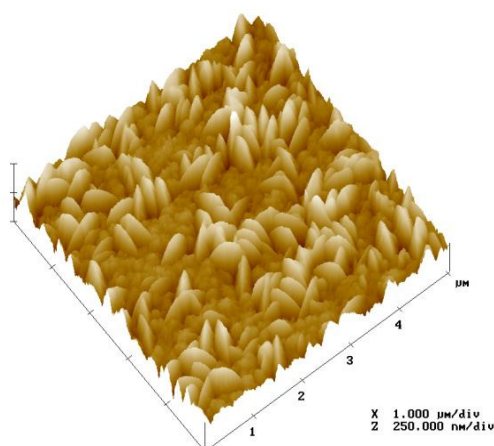- holographic and tomographic systems, …

Figure 1. AFM inspection of the surface of a 2.93 microns thick zirconium oxy-nitride film on an Inconel 738LC substrate. Tri-dimensional representation enhancing, by colour shading, the shape of the relevant surface structures.
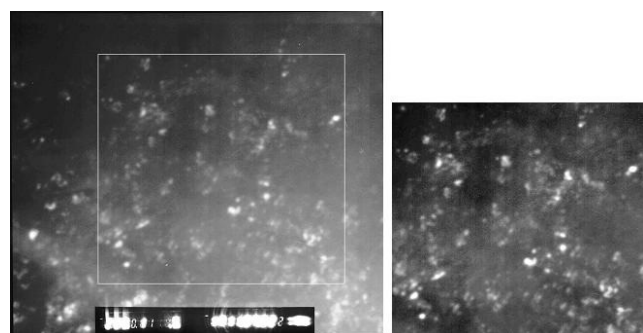

Figure 2. The proper choice of a region of interest ROI is very important in any IP procedure. In the image on the right the illumination non-homogeneity was corrected in the ROI.

2. Basic concepts

In the brief presentation that follows we shall consider the image, object of our IP, as bidimensional functions as conventionally. Furthermore we shall refer mostly to digital image processing in the Cartesian and Fourier spaces [1-13].

An image is considered to be a function of two real variables, for example, I(x,y) of brightness (grey level, colour component, …) of the image at the real co-ordinate position (x,y). In general an image may be considered to be formed by or to contain sub-images. All or some of those regions can be chosen as regions-of-interest (ROI) and processed, independently or not. This concept reflects the fact that images frequently contain collections of meaningful objects or structures. In modern sophisticated image processing system it should be possible to apply specific image processing operations to selected regions while preserving the rest of it or else to preserve the selected ROI from its neighbour regions. The definition and unambiguous location of the ROI or ROIs in the image is the first task to be executed in an IP procedure. The proper choice of the ROI according to the image any apriori knowledge and our IP objectives is fundamental to the success of the processing sought. In Figure 2 the importance of a proper ROI selection is illustrated with an example of illumination homogenization in a X ray diffraction image [15].

Image processing techniques are usually divided in two major categories: radiometric and geometrical. In geometric operations in opposition to radiometric ones the grey (colour, or any other entity n general) level in each point (pixel) of the image is changed accordingly to the image' grey values on its neighbourhood. Eventually the new grey level in one pixel can even be totally independent of its original value as it happens for instance in edge detection.

Radiometric operators act on the original images changing its brightness (colour) distribution.

In digital IP the image is represented by a 2D matrix that is processed typically by multiplication by another matrix or series of matrix sequentially.

There is a large number of well established IP operators available, from simple brightness reduction operators to complex neural network' skeletization and following operators. In virtually all IP procedures it is required the application of a series of operators in a sequential clearly established way.

Bellow we provide a list of the most important operators or processing techniques. For some of the more useful ones in surface characterisation, a brief description is provided together with a few examples of application [1-13]:

- Brightness and contrast
- Contrast stretching and histogram equalisation and manipulation (Figure 3)
- Binary operations
- Arithmetic-based operations
- Convolution-based operations in the spatial domain or in the frequency domain
- Smoothing Operations
- Linear and non-linear Filters
- Derivative-based Operators
- Morphological Operators -Dilation and Erosion, Boolean Convolution, Opening and Closing, Skeletonization, Propagation
- Morphological smoothing enhancing and edge detection (Figure 4 and 5)
- Shadowing Correction
- Defocus correction
- Enhancement and Restoration Techniques
- Unsharp masking, Noise suppression, Distortion suppression, Segmentation
- Thresholding and binarization
- Colour manipulation
- Pseudo colouring

- Processing in the Fourier space. Spatial filtering. Correlation and convolution.
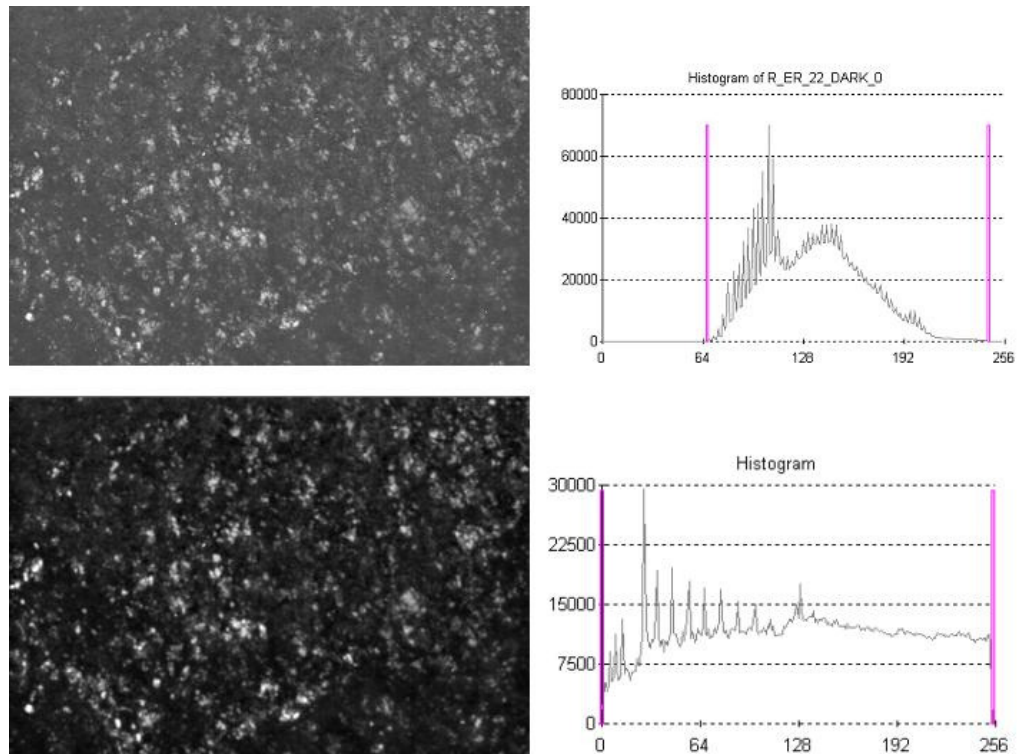- Measuring and characterisation (Figure 6 and Table 1).

Figure 3. Low contrast images (top left) can be improved by manipulation of its histogram (top right). A simple histogram stretching (bottom) gives a much clear image.
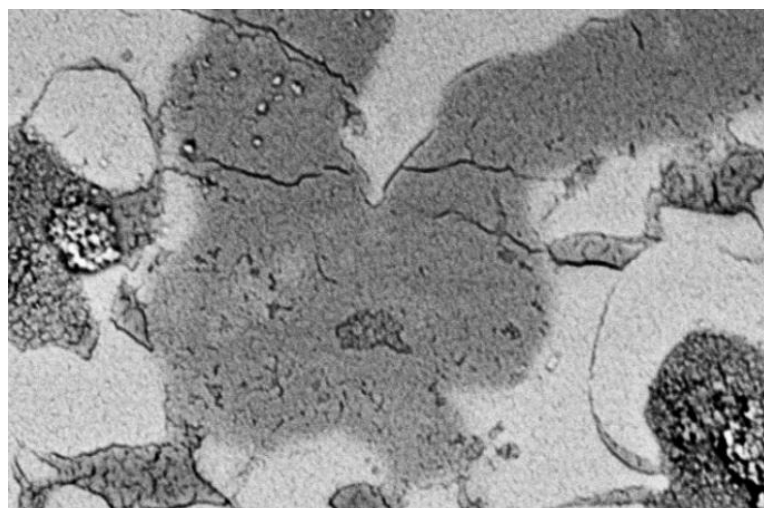

Figure 4. 22x15 μm^2 SEM image obtained at 20KeV. The SEM image show microcracks in oxide grow of a thermal barrier coating (IN738LC substrate, interlayer VPS NiCoCrAlY, top layer APS: $ZrO_2 - 8Y_2O_3$) after annealing at 1100 ºC for 100 h in air.

3. An example of IP application to the characterization of a thin film.
In this chapter the importance of IP in the characterization of thin films is exemplified in the process of measurement of the porosity of a multilayer thermal barrier coating (TBC).
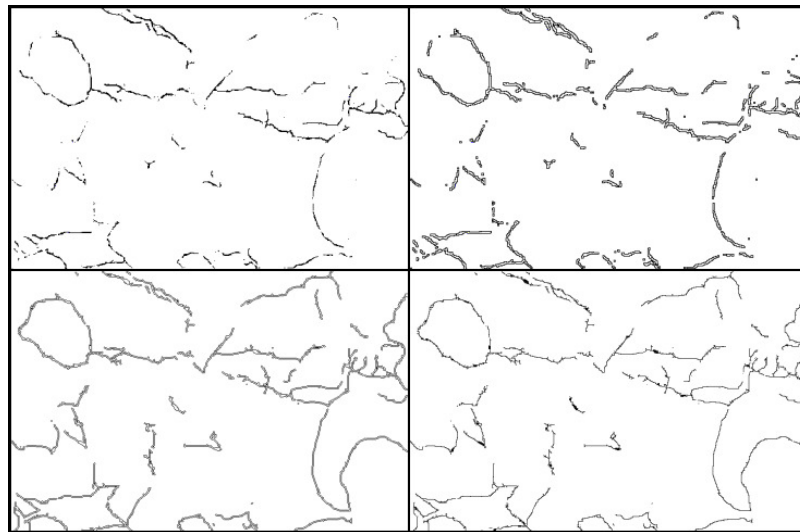
Figure 5. Results of different edge detection procedures on the micrograph presented in figure 4. The successive application of radiometric and morphological operators allows a reasonable identification of the major relevant cracks on the structure (top left image). The application of a Sobel differential operator enhances its visibility (top right). Better results can be obtained by the application of a negative Laplacian edge detector (bottom left) or even an advanced following technique using neural network processing (bottom right).
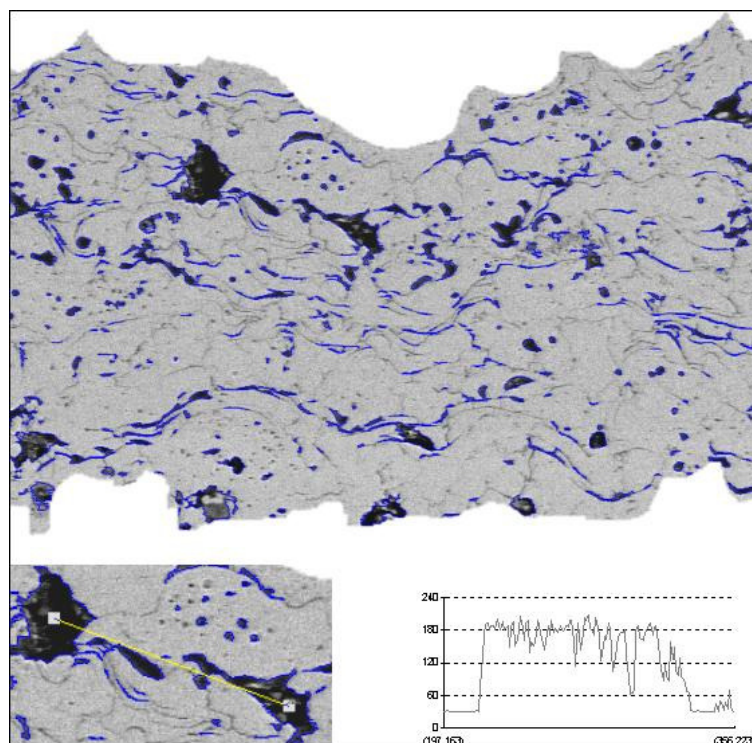
Figure 6. SEM micrograph of a $ZrO_2 - 8wt\%Y_2O_3$ coating produced by APS. The image was processed for blob analysis and one profile of a relevant zone of the image is presented (bottom).

Ceramic coatings of engineering materials such as Zirconia (ZrO2) partially or totally stabilized by oxides [16] such as Y203, MgO, or Al203 are used for a variety of technological applications requiring thermal insulation, wear and erosion resistance or protection from oxidation, sulfidation and

hot corrosion [17-19]. These kinds of coatings have been applied as Thermal Barrier Coatings (TBCs) for protection of metallic components in gas turbines (vanes, blades, shrouds, etc.) and diesel engines, and improved performance at high temperatures [20-23]. It allows increase in operating temperature and/or reducing the cooling systems needed due to the temperature gradient across the thick ceramic coating that permit better thermodynamic performance and lower emissions without requiring major alloy development. TBCs, traditionally, consist in a thick stabilized ZrO2 top coating commonly deposited by atmospheric plasma spraying (APS) on super-alloys pre-coated with a metallic bond coat (NiCoCrAlY) produced by vacuum plasma spraying (VPS). The ZrO2 top coating has a porous and laminar structure and consists of splats with cracks perpendicular to the surface, this porous structure allows the increase in the thermal isolation and the cracks permit better stresses accommodation [16, 24]. The metallic bond coat improve the diffusion of contaminants and the mismatches of the thermal expansion between top coat and the substrate and this form increase the lifetime of operation for the components.

Nowadays higher operation temperatures are required and in order to obtain systems of coatings that allow its ranges of temperatures we need develop new materials for coatings or new architectures for the existents materials. These new concepts of TBCs should have lower thermal conductivity, and to be stable at higher temperatures [21, 22].

In order to obtain better thermal isolation, in this contribution we present a new concept of TBC. It consists in a conventional bond coat and a graded ZrO2- 8 wt.%Y2O3 top coat.

Below we provide a few results of the study of structural properties of ZrO2Y2O3 multilayered coatings focusing on the porosity of the microlayers. In order to increase the efficiency of the thermal barrier the different layers will have different porosity increasing towards the surface.

The SEM images (figure 4) are processed using dedicated routines, in order to measure the porosity of the coatings [6,24,25]. Not only the porosity values for each layer were obtained but also it was evaluated the way the porosity changes along the coatings cross-section. For comparison the porosity is also determined by the conventional mercury intrusion method [24].

The SEM images were processed for analysis of the microstructure (figure 6 and table 1) and measure the thickness of each microlayer for the different porosities along cross section (table 2) of all coatings. After annealing in air all coatings presents a sintering structure and a reduction in porosity contents [23] it is also observed a thermal grow oxide (TGO) between bond and top coat.

Table 1. Characterisation parameters. Results of the blob analysis on the image of figure 6.

Label	Area	Perimeter	Convex Perimeter	Roughness	Length	Elongation	Min. Pixel	Max. Pixel	Std. Dev. of Pixels	Centroid X (Gray)	Centroid Y (Gray)
1	14	16.24	14.97	1.085	5.638	2.271	59	121	17.18	723.2	18.62
2	16	17.66	16.62	1.062	6.281	2.466	87	120	8.235	698.4	23.48
8	18	20.49	19	1.078	7.99	3.546	75	118	13.99	675.1	37.71
9	28	32.49	30.5	1.065	14.28	7.285	34	114	22.54	65.87	38.25
17	29	32.63	29.33	1.112	14.28	7.035	42	121	22.05	679.9	44.84
29	33	47.31	44.44	1.065	22.17	14.89	30	120	24.14	69.58	51.59
35	111	102.2	93.79	1.09	48.84	21.49	41	121	18.39	20.59	64.62
36	11	24.49	21.98	1.114	11.27	11.54	98	122	7.192	47.5	53.98
37	24	28.49	26.72	1.066	12.29	6.293	34	119	24.22	89	54.33
41	10	18.24	16.84	1.083	7.847	6.157	86	119	10.55	652.4	55.11
47	231	96.08	81.5	1.179	42.62	7.864	30	122	28.48	168.8	66.53
53	11	13.66	12.84	1.064	4.225	1.623	66	119	16.43	123	59.01
56	10	22.83	21.58	1.058	10.46	10.94	67	116	15.72	71.49	59.66
62	16	26.49	24.01	1.103	11.9	8.847	86	121	11.81	659.7	60.57

Traditionally the porosity in thermal barrier coatings is characterised qualitatively by microstructure observation and quantitatively by mercury intrusion porosimetry (MIP) technique besides coating density measurement. The direct examination of coatings microstructure from cross-sections of a coating using a scanning electron microscopy (SEM) gives comparative information about porosity for the different coatings. The chemical composition of the microstructure is

represented in the images by grey level variation. Pores appear very dark allowing us to distinguish and quantify them by image analysis. By this method we can not obtain information about the 3-D pore network or connectivity between them [24,25]. For our analysis two series of SEM images one with 400X magnification and other with 500X magnification were acquired.

Using the MIP it is possible to obtain measurements of total porosimetry for open pores and the evaluation of pore size distribution. MIP does provide information about the connectivity of the pores and microscopy reveals information about pore geometry, so have a big interest in to combine these two techniques for a more complete analysis.

The digital micrographs were evaluated on a Matrox II program for image analysis.

The pores were identified by thresholding the brightness of the pores to produce a binary image. The dark area fraction in the binary image was evaluated and the percentage calculated [6].

The corresponding porosity values for the different coatings are presented in Table 2.

Table 2. Coating porosity measured by image analysis and Hg-porosimetry

Samples	HP	GPI	GPII	GPIII
Hg –Porosity (%)	14.75	15.31	15.29	13.38
Image Analysis (%)[a]	11.79	13.08	15.48	13.34
Image Analysis (%)[b]	11.15	10.76	12.73	9.34
Image Analysis after annealing (%)[a]	8.44	8.33	9.90	10.57
Image Analysis after annealing (%)[b]	6.94	7.99	7.40	8.27

[a]Porosity with small cracks and ribbons.
[b]Porosity without ribbons.

We can see in Table 2 a considerable difference between the measured Hg-porosities and the porosities evaluated by image analysis. Also has been seen a reduction in porosity values after annealing for all samples, its reduction is mainly due the sintering effects [26,27]. Hg-porosimeter gives reasonable results for the measure of small pores and microcracks but it fails for pores with radii larger than 60 μm. On the other hand image analysis give acceptable results of porosity due to the contribution of small pores and small microcracks between lamellae within the plasma-sprayed coatings and the big pores are measured easily. In agreement with SEM analysis and deposition parameters we estimate the thickness of each microlayer in the graded coatings and by image analysis was determined the porosity values of each layer. The porosity increase from the interface with bond coat to the surface and it is noticed a decrease in porosity values for higher values of power deposition and a increase with an increase of the working distance. For the sample with constant deposition parameters we observe a little decrease in the porosity values to the surface that is justified by increase the substrate temperature during deposition.

4. Conclusions

A major increase in the power and sophistication of modern computing with the implementation of optical computing and distributed and parallel processing will boost the application of image digitalisation and processing including in real-time situations. An extended concept of image above the traditional optical definition is already accepted in this field. The processing of bi (or even multi) dimensional functions will allows us humans to perform easier and more effective inspection and evaluation tasks. The gap between the efficiency of our visual systems and those of future imaging and image processing machines will be steadily closed given us even more valuable efficient non invasive automated inspection and diagnostic tools in nano sciences and technologies.

The work herein proved the usefulness of image processing in the qualitative and quantitative characterization of the porosity of thin multilayer thermal barrier coatings (TBCs).

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