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SEGMENTATION AND GRAIN SIZE OF CERAMICS

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ABSTRACT

This paper presents some methods to automatically extract the grain boundaries of materials in order to develop an automatic method to determine the grain size and morphological parameters of ceramic materials. Results are presented in the case of sintered cerine (CeO_2) materials.

Keywords: cerine, granulometry, mathematical morphology, segmentation.

INTRODUCTION

There is a direct relationship between the fabrication process, the microstructure and the properties of materials (Chermant, 1989; Tenckhoff and Vöhringer, 1990; Rice, 1997; 1998; 2000). As far as ceramics are concerned, the control of the microstructure all along the fabrication process is of critical importance. Indeed, ceramics are brittle and are accordingly very sensitive to small defects and to the presence of very large grains whose presence initiates the rupture under stress solicitation. Moreover, a variety of physical properties depend on grain size $\overline{D}$, for example, the yield stress of a material is a function of $\overline{D}^{-1/2}$.

By now, the mean grain size of a ceramic is almost always determined either manually or semi-automatically, using furthermore a too small number of grains (Bennett *et al*., 1997). Automatic methods of image analysis are to all intents and purposes statistical methods, and will provide the correct result if all the grain boundaries are revealed; this is never the case.

The scope of this paper is first to use classical metallographic methods to reveal the maximum of the grain boundaries. Second, it is to use and/or develop methods of segmentation based on mathematical morphology (Serra, 1982) and linear filters that will complete the grain boundaries detection and give access to classical morphological parameters. Ultimately, it will be possible to model such microstructures using probabilistic models and then, to investigate the sintering both from the changes in the stereological parameters and from the model.

As sintering investigations of ceramics concern essentially alumina, silica, magnesia or anatase (see for example Kingery *et al*., 1976), we have selected for the present investigation a less common monolithic ceramic, the cerium oxide CeO_2; however, all these results can be transposed to any other types of ceramic or to granular materials.

MATERIALS AND METHODS

Sintered materials were prepared from a CeO_2 powder 99.95% purity (Rhône-Poulenc), with a specific surface area of 2.7 m^2/g. Then, 0.1% of TiO_2 and a binder (polyethylene glycol, PEG 300) were added to the CeO_2 powder. The mixture was attrited, atomised, sieved and then compacted in a cylindrical mould. The achieved samples were 20 mm in diameter and 4 to 6 mm thick, as it is often the case in classical methods of powder metallurgy (see for example Lenel, 1980).

The materials were sintered in air using different time-temperature cycles (temperature range: 1100°C to 1450°C; time range: 6 min and 20 h) (Arnould *et al*., 2000). After sintering, the compacts were polished using classical techniques of metallography with diamond pastes on hard clothes up to 0.06 µm.

To reveal the microstructure we have used both, plasma etchings in $O_2 + CF_4$ atmosphere and thermal etchings (March MPS 300 Super Plasmod equipped with the March GCM-200 modulus, Concord, USA), (Herb, 1989). The best results were obtained by a thermal etching performed at temperature 50°C lower than the sintering temperature and during 5 min to 15 min.

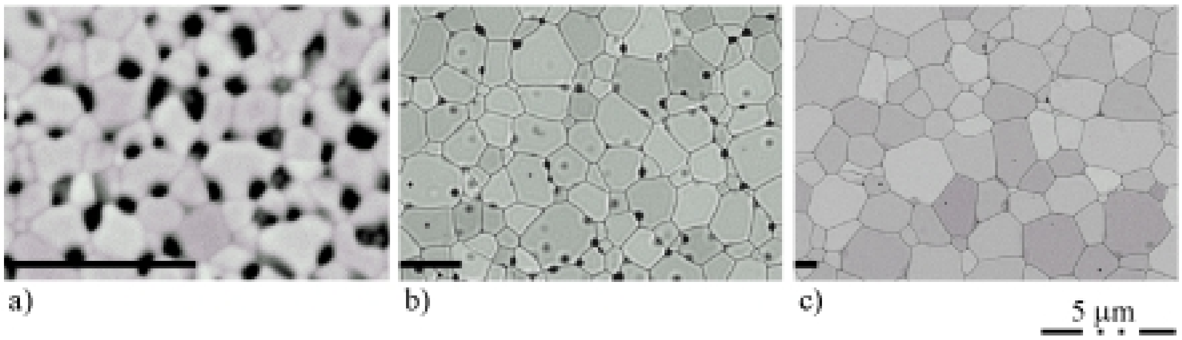

Fig. 1. *Microstructure of CeO_2 sintered 2 h at: a) 1200°C; b) 1300°C; c) 1400°C. Full bars at the bottom of the micrographs correspond to 5 µm.*

Microstructures were observed by scanning electron microscopy SEM (Jeol JSM 6400). For every sample, 50 images were acquired and automatically analysed using the image analysis Aphelion software (ADCIS) and more specifically mathematical morphology (Serra, 1982; Coster and Chermant, 1989). Examples of microstructures are presented in Fig. 1.

IMAGE PROCESSING: SEGMENTATION OF GRAIN BOUNDARIES

As it can be seen in Fig. 1, all the grain boundaries are not revealed by the thermal etching. So one must use or develop methods of segmentation. The main difficulty in the segmentation procedure is to avoid over- and/or sub-segmentation.

Over-segmentation concerns samples where defects are evidenced by the SEM: they are due either to impurities introduced during the fabrication process (or they are just intrinsic to the materials), to crevasses appearing with a non homogeneous etching, or to some pores badly detected due to the classical phenomenon of electronic discharge as the material is not conductive (Fig. 2a).

When all the microstructure is not completely revealed whatever the magnification is, or when the microstructure is very fine (as it is the case for very porous materials) which requires to use high magnification during the acquisition, one is faced with the reciprocal problem of sub-segmentation: the images present grain boundaries with grey tone levels very close to that of the grains themselves. So one detects only partially the grain boundaries, *i.e.*, only some disconnected lines are revealed.

One must separate the investigated materials in two classes, depending on their densification. For dense materials, classical filters can be used without problems, as the images are acquired at low magnification. The black top-hat (Meyer, 1978) is applied to the grey tone functions, yielding all the outlines of the grain boundaries, and is followed by a threshold of the pores; then a skeleton is used and the pore image is added (Fig. 3). In some cases, the result is improved when a high pass and/or a low pass filter(s) are first applied. The high pass filter enhances the lower outlines, while the low pass filter eliminates the noise. It is the combination of these filters which leads to correct results.

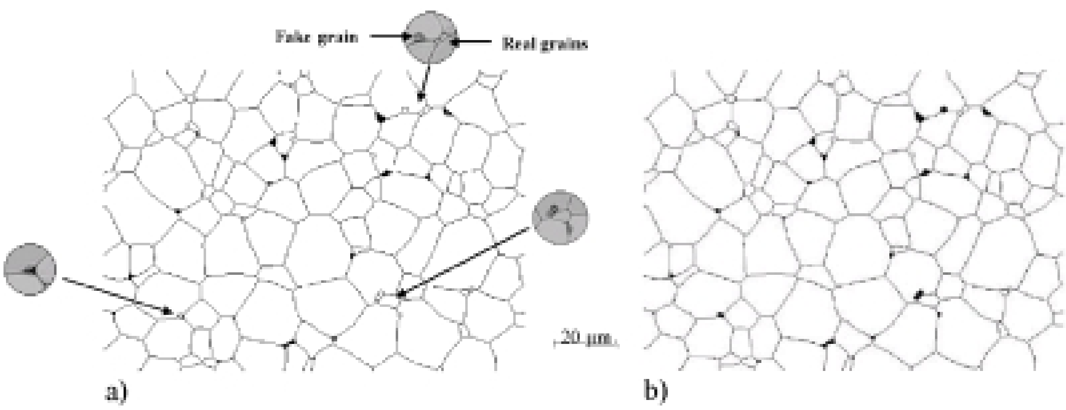

Fig. 2. *a) Image presenting some small real and fake grains in CeO_2 samples; b) result after the use of specific filters which will be described latter on.*

In this first class, the problem of oversegmentation may be important in some samples. It is the combination of two filters that will eliminate the fake grains: i) the first one allows to eliminate all the grains having two neighbours, as this is physically not admissible; ii) the second filter uses the grey tone level to differentiate the fake grains from the real ones, as a real grain shows generally outlines finer than those of a fake grain and as the grey tone level is higher (Fig. 2b).

Although less common in this class of materials, the problem of sub-segmentation may be encountered. The "conical" dilation (see the scheme in Fig. 4, (Serra, 1988)) is well adapted: the extremities of the partial segments are recovered, and dilated by a cone oriented in a given direction until the two parts of skeleton are connected (Fig. 4).

For very porous materials, corresponding to the second class of materials, extracting the grain boundaries is even more complex. The previous filters and their combination are not appropriate leaving the problem of sub-segmentation unsolved. The conical dilation would also fail due to the noise of the images: in fact, it is very difficult to isolate the end points of the partial grain boundary independently on the end points due to the skeleton of residual noise. In this second class of materials, the best results are obtained using morphological gradients (Meyer, 1992) allowing to recover a certain number of outlines. As an example, the external gradient is the residue between the dilated and the initial image.

When all the grain boundaries cannot be detected, the watershed segmentation can be used as a complementary transformation. In the classical watershed, where the segmentation of the image starts from the local minima of the grey tone image, one would obtain an over segmentation. Here, the point is to start from other markers selected as a function of the problem to solve (Beucher, 1982; Beucher and Meyer 1992). Correct results were obtained by using the morphological gradient image filtered by a low-pass (weighted mean obtained by a kernel of convolution of size from 3×3 to 7×7). But the difficulty lies in obtaining all the desired markers (in other words, only one marker per physical grain).

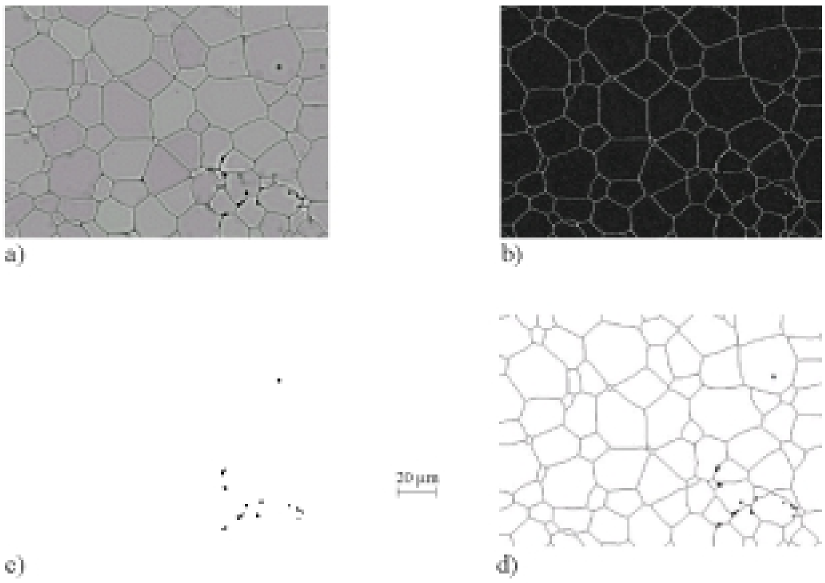

Fig. 3. *Transformations used after an over-segmentation: a) initial image (CeO_2, 1400°C, 1 h); b) effect of the black top-hat; c) pore threshold; d) effect of the threshold, with addition of the pore image and the skeletonization.*

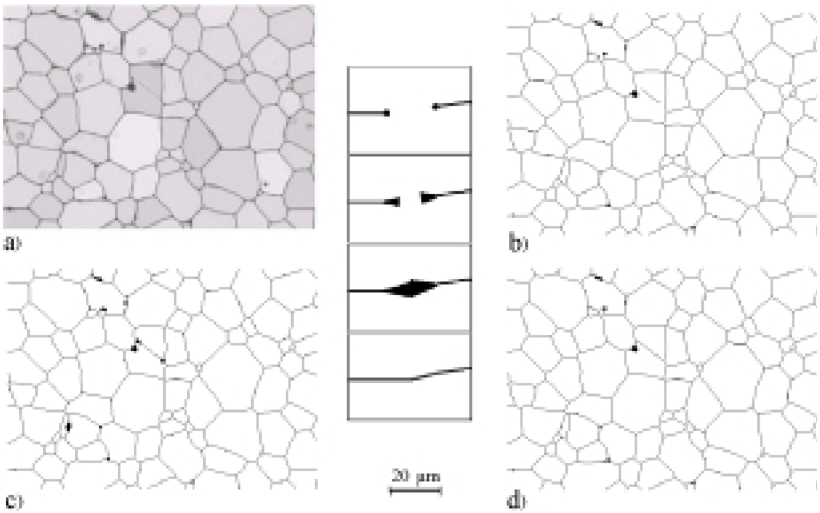

Fig. 4. *Filter used for a sub-segmentation: a) initial image (CeO_2, 1450°C, 2 h); b) skeletonization; c) "conical" dilation of the end points; d) final image.*

RESULTS

The different strategies of segmentation presented above have permitted to obtain automatically the morphological parameters of these sintered materials. Very accurate results were obtained as in the best case and worst case one obtains an error of 0.2% and 5%, respectively. Here are our preliminary results.

For example, Fig. 5 shows the granulometric spectra, obtained by individual analysis, for CeO_2 sintered at 1450°C during 6 min to 5 h. It is in agreement with the classical results on sintering: there is a grain growth with time at a given temperature, in our experimental conditions from 2 µm to 15 µm.

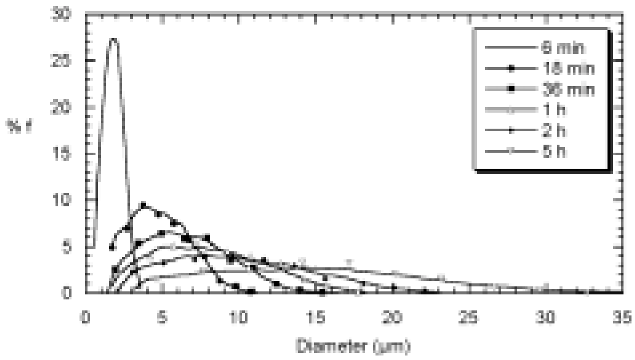

Fig. 5. *Granulometric density of CeO_2 grains sintered at 1450°C during 6 min to 5 h.*

One has also followed the change in the mean number of neighbours (Fig. 6): it remains close to 6, whatever the sintering time is. This result corresponds to a granular system in a state of equilibrium. This morphological parameter is of main importance in order to model such microstructures by probabilistic models.

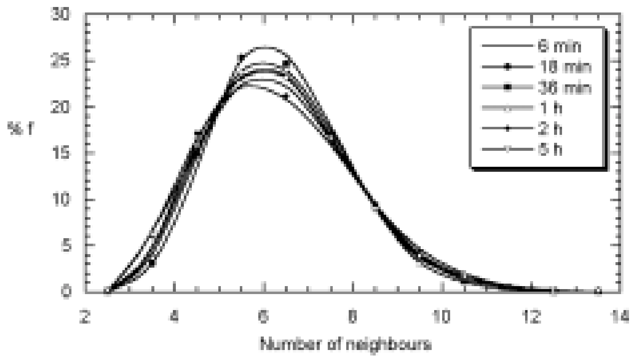

Fig. 6. *Granulometric density of the neighbours of given grains for CeO_2 sintered at 1450°C during 6 min to 5 h.*

CONCLUSION

In this work, we have used the main methods of morphological segmentation to obtain fully automatically the granular structure of the cerine: top hat and watershed segmentation. To complete the segmentation, directional dilation of incomplete grain boundaries was used before applied watershed process. The innovating aspects of such a segmentation are based on combinations of classical and appropriate filters. The proposed segmentation and filtering are really appropriate for these materials and permit automatic measurements. The watershed and the top hat procedures are very adaptive methods through the choice of the parameter (top hat) or the marker (watershed) and produce accurate segmentations.

These methods can be easily transposed to other ceramics or to any granular materials. The segmented images can be analysed by classical or morphological methods to obtain, for example, granulometries or topological datae.

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