

TRENDS AND CHALLENGES IN THE MORPHOLOGICAL STUDY OF CERAMICS

Michel COSTER, Jean Louis CHERMANT

LERMAT, UPRESA 6004, ISMRA, 6 Bd Maréchal Juin,
14050 CAEN CEDEX France

SUMMARY

Ceramic materials are always fabricated from powders or mix of powders. Their solid state is obtained most often by sintering. Ceramic materials can be classified in two categories: monophased or polyphased ceramics. Cermets (ceramic-metal) are generally obtained by liquid phase sintering; they are made of a metal matrix where a ceramic phase is dispersed.

The study of the morphology of these materials involves, first, the use of a metallographic technique to reveal the microstructure by an appropriated etching.

The next step consists of isolating phases or grains of material by segmentation and threshold methods. Each method has to be adapted to the analysed material.

Then, one proceeds to the analysis itself. Classically, accessible stereological parameters in the R^2 space are measured. They allow to estimate corresponding parameters in R^3 . A finer parametric analysis requires a characterisation of the shape, the anisotropy, the state of dispersion and mainly the size, using various methods (granulometry, granulomorphy, covariance, ...). For these analyses, mathematical morphology will appear as one of the most efficient tool for morphological purpose.

Finally one will show that the use of probabilistic models is an interesting way to characterise, by a simple but complete manner, the microstructure of ceramics. Their conditions of application to real microstructures and corresponding tests will be specified.

Key words: ceramics, image analysis, mathematical morphology, probabilistic models, stereology.

INTRODUCTION

The ceramic area

A ceramic can be defined, for example, as an inorganic and non-metallic material. It is elaborated by a thermal process at high temperature. This definition includes glasses. Traditional ceramics are generally manufactured from clays.

The new ceramics are not always easy to be classified. One can however establish a non-exhaustive list of the most known: oxides, carbides, borides, nitrides, titanates, ... and also ceramic composites. Bulk ceramics are often made from classical process techniques

(hot-pressing, casting from slurry, extrusion). Contrarily to traditional ceramics, the densification is not always obtained thanks to a binder vitreous phase. Often the densification results from a process of solid phase diffusion under the effect of the temperature in agglomerate of fine grains. This thermal process corresponds to the sintering.

By definition, metals are out of the ceramic area. Nevertheless, there is continuity between these two classes with cermets, obtained by liquid phase sintering applied to two mix powders, one metallic and the other ceramic. Ceramics present fundamental properties: thermal and chemical inertia, high mechanical strengths at high temperatures compared to classical metals or alloys. But they are generally brittle. They are often insulator or semiconductor. In the framework of this paper, we will limit the study to the bulk ceramics during and after the sintering.

Relationships between physical properties and morphology

Before to define methods and parameters allowing to characterise ceramics, it is necessary to understand the dependence of physical properties with the microstructure (one can quote Rhines (1986) or Exner and Hougardy (1988)). One can say that three important factors are concerned regarding their properties : i) the first factor is the variety of morphologies in ceramics; ii) the second factor corresponds to the notion of heterogeneity in a wide sense, (Stoyan and Schnabel, 1990); iii) the last factor is related to the quality of the interface between the grains. Image analysis is a tool well adapted to study the two first factors. The study of the homogeneity of a microstructure requires more complex operations. But in some cases classical methods are unusable. Thus some properties, as the toughness, depend especially on extreme morphological characteristics, largest grains for example (Virkar and Gordon, 1977) and therefore rarely present. In these conditions, analysis of classical images seems not suitable. Finally, the third factor is completely far from the area of image analysis since it concerns the characterisation of the structure and the composition of interfaces between grains at the nanometric scale.

CERAMOGRAPHIC METHODS

Grinding and polishing

Preparation of polished sections of ceramics is different from those conventionally employed for metallic materials because their toughness is very low and they are generally very hard materials. A coarse grinding stage produces considerable amount of microcracks. A grinding with abrasive powders of size smaller than 30 μm during long enough times give good results. The final polishing is generally obtained in using diamond paste of approximately 1 μm . Guidelines on how to choose a grinding method may be found in Hubner and Hausner (1983) or in Carle (1991). Procedures to polish cermets are closed to that for metals because they are less weak than ceramics.

Etching

After the polishing, the two main techniques of etching to reveal the ceramic and cermet microstructures are thermal and chemical etchings. The choice of the process depends on the nature of the material and the mode of observation (generally optical or scanning electron microscope). Over-etching is to be avoided, since it can modify the appearance of the microstructure.

Thermal etching, most often used for oxide ceramics, can reveal clearly the grain boundaries, but there is always a risk to modify the microstructure. The maximum temperature for the thermal etching should be at least about 150K below the temperature of sintering. The

composition of the atmosphere (reducing or oxidising) and especially the potential of oxygen has a great importance for the thermal etching, (Charollais, 1997). In the case of monophasic polycrystalline ceramics, it may be necessary to use more severe etching for SEM images than for optical images to obtain good contrast at grain boundaries, (Figure 1a).

The chemical etching of oxide ceramics is often difficult to control and to reproduce. To reveal oxide microstructure, one uses generally a chemical solution, which gives soluble complexes. Moreover, the colour can vary according to the crystallographic grain orientation; so the revelation of grain boundaries is often incomplete, (Figure 1b). The automatic image segmentation is then very difficult, but can be developed, as in the case of WC-Co (Gauthier *et al.*, 1996). For carbides and cermets, one uses an alkaline solution (soda or Murakami's reagent). Petzow (1979) and Carle (1991) have given bibliographic lists of etching methods for many materials.

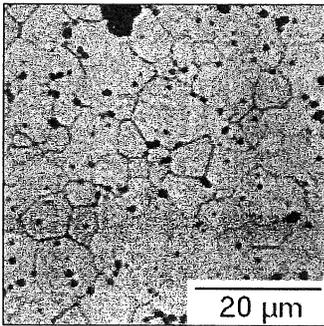


Figure 1a : Optical micrograph of sintered UO₂ after thermal etching.

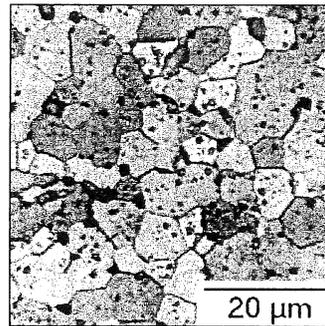


Figure 1b : Optical micrograph of sintered UO₂ after chemical etching.

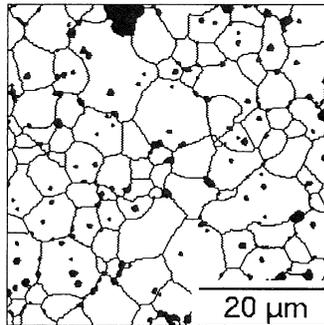


Figure 2 : Segmented binary image of Figure 1a by image processing.

IMAGE SEGMENTATION

Of course, it is impossible to propose a general method to segment images. The choice of an adequate method depends on several factors: mode of observation (optical, SEM, TEM, confocal, ...), number of phases, type of etching, topology of the microstructure, parameters to be measured. We will limit, in the following, our examples to two great classes of ceramics: polycrystalline materials, more or less porous, and cermets.

Polycrystalline and monophased ceramics

One must, first, recall that image analysis of polished section does not allow to follow all the different steps of the sintering route. Indeed, at the beginning of the sintering, the material is always very porous and chemical or thermal etching does not give good results. In these conditions one can characterise, easily, only the porous phase on polished sections. The morphological study of the granular structure can be only undertaken if the material is enough dense (porosity < 15%).

Threshold of the porous phase

Automatic threshold of the porous phase is generally very easy. One can use a method based on the grey level histogram. The threshold by maximisation of entropy gives good results on SEM images when the porosity is sufficiently small, (Zacharie, 1997; Coster *et al*, 1997). When the porosity is more important, the automatic choice of the threshold can be obtained from the analysis of the gradient of the grey level histogram.

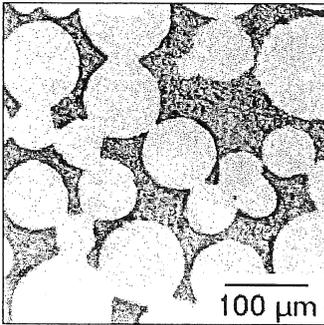


Figure 3a : Image of sintered glass obtained by optical microscopy.

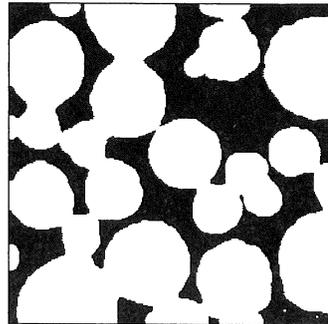


Figure 3b : Binary image of the figure 3a obtained after an opening followed by a threshold.

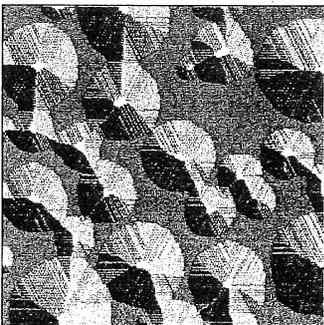


Figure 3c : Distance image of the figure 3b with h dilated minima as markers.

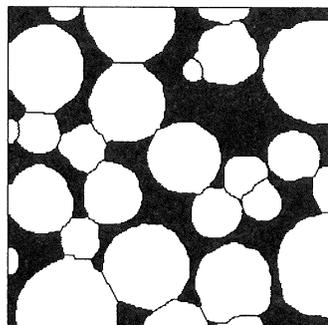


Figure 3d : Segmented image obtained from watershed processing on the figure 3c.

Segmentation of grains boundaries with low porosity

Sintered ceramic microstructures present always intergranular residual porosity. This porosity is generally darker than grain boundaries revealed by thermal etching. In these conditions, the segmentation of images will be therefore made in two steps : i) with a first threshold, one

selects only the porous phase; ii) then this porous phase is filtered. In the case of UO_2 (Charollais, 1997), one proceeds to the filling of pores, then to a reconstruction by opening to eliminate artefacts on this first binary image. A higher threshold allows to preserve grain boundaries. A SKIZ (skeleton by influence zone) allows to reduce the thickness of grain boundaries to one pixel. Union of these two images gives the final binary image, (Figure 2).

Segmentation of grains boundaries without etching

When the porosity is enough important, thermal or chemical etching methods does not allow to reveal the granular structure. In these conditions, it is necessary to make hypotheses on the shape of the grains. If these grains are convex and relatively circular, a watershed segmentation of the distance image is possible, (Beucher and Meyer, 1992). This method has already been used in the case of sintered metals. An illustration is then presented in the case of a sintered glass (Figures 3a, 3b,3c, 3d).

Cermets

Cermets with "circular" grains

Cermets with "circular" grains can be processed as sintered glass. In the case of the use of a SEM with EDS analysis, one will be able to have X images of the main chemical elements of each phases to quantify the morphology.

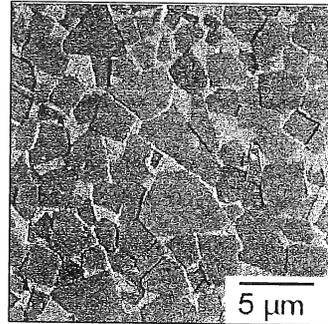
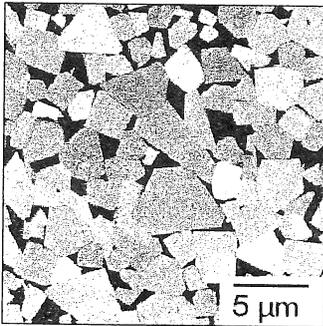


Figure 4a : SEM backscattered electron image of WC-Co cermet. Figure 4b : SEM secondary electron image of WC-Co cermet.

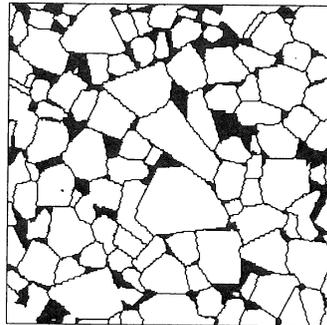


Figure 4c : Segmented image of WC-Co cermet.

WC-Co cermets

WC-Co cermets are more difficult to segment because grains are polyhedral. For this segmentation, Gauthier *et al* (1996) have proposed to use multimodal SEM images (image by backscattered electrons linked to the composition (Figure 4a), and image by secondary electrons linked to the topography (Figure 4b)). These images give indeed complementary information. From the composition image, one extracts the cobalt phase by an automatic threshold (maximisation of the interclass variance). A part of grain boundaries is recovered. Then, one uses the topographic image and one applies linear openings and closings followed by a top-hat transformation in the perpendicular direction (Serra, 1982; Coster and Chermant, 1985; 1989). An automatic threshold by maximisation of entropy allows to extract other grain boundaries. Then, one undertakes the union of the cobalt phase with the already reconstructed grain boundaries. Boundaries non revealed are partially completed by directional morphology (Kurdy and Jeulin, 1989). The distance image is then created and missing boundaries are obtained by applying a watershed to the image of distances (Beucher and Meyer, 1992), (Figure 4c). The result of the segmentation has been compared to a manual segmentation. The measured diameter gap does not exceed 1% as compared to the reference.

PARAMETRICAL CHARACTERIZATION OF THE MICROSTRUCTURE

Ceramics can most often be considered as two phase systems : i) pore and ceramics phase; ii) matrix or refractory phase. Parametric characterisation methods of the microstructure of ceramics can be divided in two categories. If a segmentation of the granular phase has not been made, one will use first classical stereological parameters. A linear granulometric analysis will give access to a complete information from the measurement of the $P(\ell)$ function (Haas *et al.*, 1967). If a segmentation of grains is applied, an individual granulometry or shape analysis can be obtained.

Stereological measurements and granulometries on non segmented images

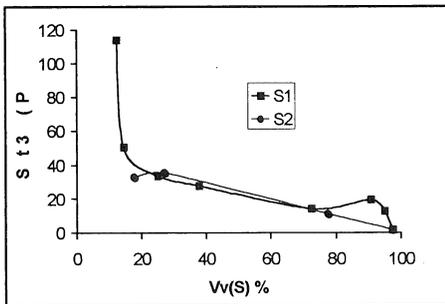


Figure 5 : Evolution of the star function with the volume fraction of solid as a function of the milling conditions (S1, S2) for BaTiO₃ sintered materials.

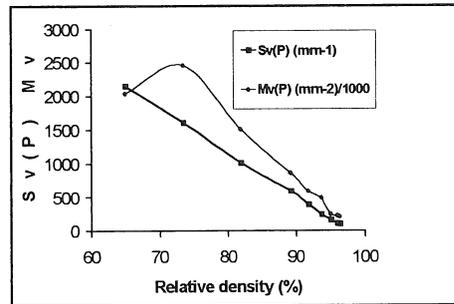


Figure 6 : Evolution of the specific surface area and of the mean specific curvature of porous/solid interface as a function of the relative density for UO₂ ceramics.

The sintering of barium titanate (BaTiO₃) has been analysed, for example, from stereological parameters in the porous phase, (Coster *et al*, 1996; 1997) : specific surface area, $S_v(P)$, mean length of pores, $L_1(P)$, integer of mean curvature, $M_v(P)$, and star function in R^3 , $St_3(P)$, determined from the $P(\ell)$ function performed on binary images of polished sections. Figure 5,

corresponding to the evolution of the star function with the volume fraction of solid, exhibits different behaviours when milling conditions change (samples S_1 and S_2). In the case of S_2 sample the strongly decreasing value of $St_3(P)$ corresponds to the closure of the channels between agglomerates.

In the same way, the evolution of the porous phase of UO_2 during the sintering has also been followed by stereological analysis (Figure 6) and the granulometry of pores by the $P(\ell)$ function, (Charollais, 1997).

Granulometries on segmented images

In the case of monophased polycrystalline ceramics, manual or automatic segmentation allows to perform an individual analysis. One can thus access to granulometries, but also to shape, anisotropy or neighbouring (topology) analysis.

If one makes a bibliographical study in the domain of the structural ceramic characterisation, it is essentially the size criterion which has been studied.

As the image is composed of grains, two main methods can be used. The first one performs granulometric operators simultaneously to the totality of grains, (morphological methods). The second one is more classical, since the granulometric sorting is made grain by grain by individual method.

Size analysis by morphological methods

The granulometry of a segmented phase can be made by morphological openings. As in the case of non-segmented images, linear granulometries can be used. Granulometries by opening with an 2D structuring element are used : i) morphological opening (erosion + dilation), ii) reconstruction opening. The bias introduced by the field of measurement is corrected by the application of the theorem of the mask of measure (Serra, 1982; Coster and Chermant, 1985; 1989). The size criterion is the size of the structuring element. Granulometries are always given in measure. Figure 7 illustrates an opening in the case of a sintered material.

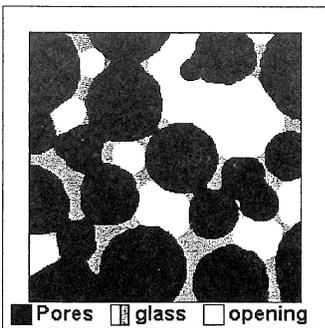


Figure 7: Example of opening of size 20 on sintered glass with respect to the theorem of the mask of measure.

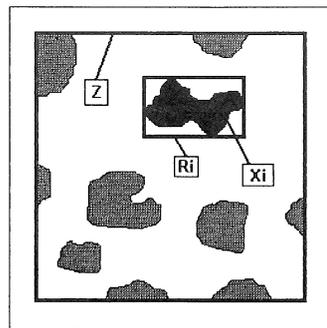


Figure 8: Correction of Miles-Lantuéjoul for parameters measured by individual method :

$$\text{Weighted coefficient } K(i) = \frac{A(Z)}{A(E^{Ri}(Z))}$$

Size analysis by individual methods

More classically, the granulometry of the segmented phase can be made by an individual analysis method. In these conditions, it is necessary to eliminate grains cutting the frame of measurement. The bias so introduced is corrected by allocating to each grain a weight inversely

proportional to its probability of inclusion in the frame of measurement by the corrective method of Miles-Lantuéjoul, (Lantuéjoul, 1980), (Figure 8). With this type of analysis, one has a great variety of size parameters, (surface area, perimeter, mean chord length, maximum and minimum Feret diameters, ...). The most robust parameter is the surface area.

Anisotropy study

Anisotropy study on non-segmented image

Sintered ceramics under uniaxial pressure give generally anisotropic structures. When the axis of compression is known, it suffices to measure the intercept according to parallel and perpendicular directions to this axis. The ratio of these intercepts defines the degree of anisotropy. This degree of anisotropy has been, for example, estimated in the case of the nuclear fuel MOX (Charollais, 1997).

Anisotropy study on granular structure

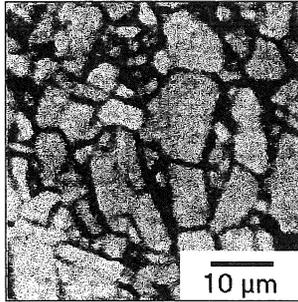


Figure 9a : Micrograph of polycrystalline SiC.

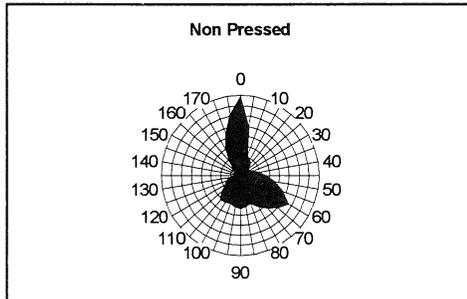


Figure 9b : Rose of directions of polycrystalline SiC.

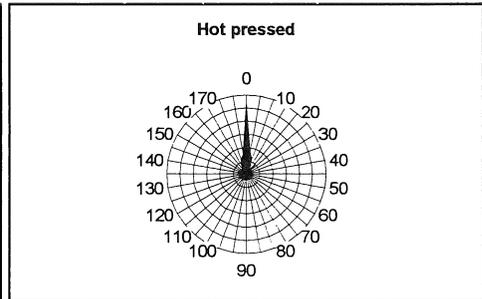


Figure 9c : Rose of directions of hot-pressed polycrystalline SiC.

The sintering of ceramics can be obtained by hot-pressing. In this case, creep phenomena is added to diffusion phenomena. When the applied pressure is not isotropic, the obtained microstructure can be anisotropic. In the case of a monophased material as sintered silicon carbide (Figure 9a), the study of the anisotropy can be made by two different ways, (Duval-Rivière *et al*, 1995). One can study the anisotropy field by field, or grain by grain. The study has been made by using the elliptic model of Serra (1982) and the rose of directions. The effect of the mechanical deformation is obtained by the study of the anisotropy before and after deformation. Table I illustrates results field by field and figures 9b and 9c show the rose of directions for analyses grain by grain. Grains are always relatively lengthened but they are

oriented more or less randomly without the effect of the pressure. They have also tendency to be aligned with the pressure.

Table I. Results of field analyses for annealed and deformed samples. Mean free path of grains $\langle L \rangle$, eccentricity e , d_1/d_2 ratios and α , are given for four SiC samples.

samples	strain ϵ_f	$\langle L \rangle$ (μm)	e	d_1/d_2	α ($^\circ$)
1	0	3.01	0.40	0.92	26
2	0.250	2.86	0.68	0.73	4
3	0	2.58	0.30	0.95	23
4	0.428	3.12	0.79	0.61	1.5

The eccentricity values of the two non deformed states (sample 1 and 3) are equivalent and correspond to a quasi-equiaxis structure for which the d_1/d_2 ratio would be equal to 1. For the two deformed specimens 2 and 4, d_1/d_2 ratios calculated from the eccentricity values are 0.73 and 0.61 respectively. Moreover grains of the two deformed specimens (2 and 4) tend to be aligned perpendicularly to the compression axis. This is indicated by the α values found close to 0° . Indeed α represents the angle between the large axis and the direction normal to the external stress σ .

Neighbouring analysis

The evolution of the distribution of the number of neighbours of a grain in R^2 during the sintering of UO_2 allows to see if the granular growth is normal (Charollais, 1997). To obtain this distribution, an automatic method on images with boundaries thinning by SKIZ is used. Each grain is isolated then dilated to cover neighbour grains. A reconstruction following by a count allows to obtain this number.

HOMOGENEITY STUDY

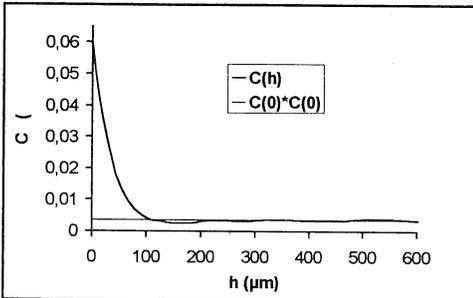


Figure 10 : Covariance of pores in UO_2 ceramic.

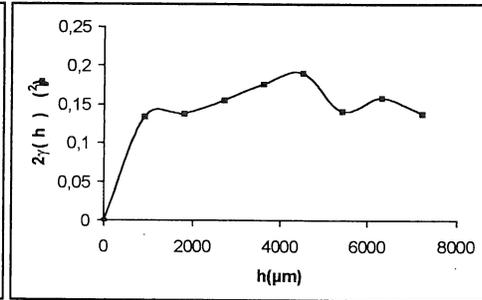


Figure 11 : Variogram of polycrystalline UO_2 obtained from the mean diameter.

The covariance has allowed to verify the good magnification. Figure 10 shows the evolution of the covariance of pores in UO_2 ceramics. The covariance reaches a plateau for a distance corresponding to 1/10 of the size of the field of measurement. To study the homogeneity of a structure, one can calculate a regularised variogram given from the equation [1].

$$2\gamma(h) = E\{[f(x+h) - f(x)]^2\} \tag{1}$$

In the case of sintered UO_2 , this variogram has been calculated from the average grain size on each field spaced by a regular manner (Figure 11). In this case, one notices that the structure reaches rapidly a plateau and therefore that it is homogeneous.

MODELLING

Stereology brings an often-satisfying reply to estimate 3D parameters. However topological parameters in R^3 , dispersion of phases, ... are not accessible from measures in R^2 . Probabilistic models bring an elegant solution to this problem (Jeulin, 1997; Jeulin *et al.*, 1995). It allows, for example, to access to the number of particles per unit volume, without any hypothesis and without using serial sectioning. Two examples on ceramics will be presented.

Two phased nuclear ceramic

Very schematically, the process to obtain the MOX can be divided in two steps: at first a rich powder mix in oxide of plutonium (25%) is performed then dilutes in a powder of uranium oxide. The final dilution allows to have a content in plutonium oxide of about 3%. The alpha autoradiography of the sintered material reveals the mesostructure of the MOX: rich plutonium islands dispersed in a matrix of UO_2 . Considering the compression axis, these islands have an ellipsoidal shape due to the applied uniaxial pressure during of the sintering, (Figure 12a).

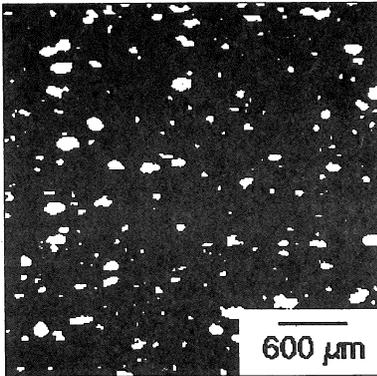


Figure 12a : Mox autoradiography image.

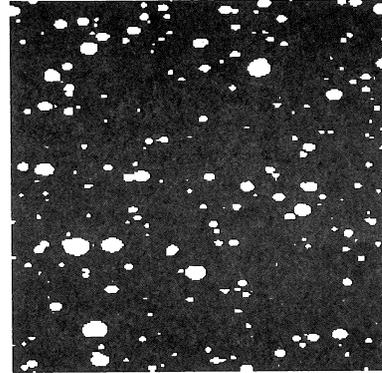


Figure 12b : Simulated Boolean model with elliptic primary grains.

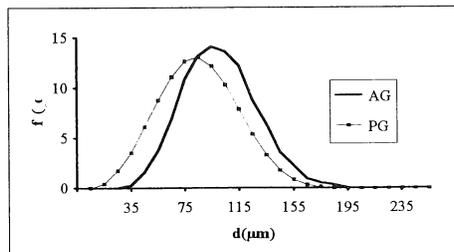


Figure 13: Distance distribution between the first closer α emitting islands (AG) and between primary grains (PG) for MOX ceramic.

Linear analyses in the direction of compression and in an orthogonal direction have allowed to calculate the anisotropy parameters. An anamorphosis was used for this the structure. Spherical primary grains describe then the anamorphosed Boolean structure. As a Boolean model remains Boolean by anamorphosis, a Boolean model with ellipsoidal grains corresponding to a log-normal distribution (Figure 12b) describes perfectly the MOX, (Charollais *et al.*, 1997). Stereological parameters of the mesostructure, such as the number of plutonium islands by unit of volume, have been estimated. A tridimensionnal structure corresponding to this model has been simulated. It allows to reach the distribution of distances between plutonium (figure 13). This last point is important to know and can be introduced in codes of calculation on the fuel.

WC-Co cermets

WC-Co cermets obtained by liquid phase sintering present polyhedral carbide grains, (Figure 14a). Several models using Poissonian primary grains have been tested: a two coloured Poisson mosaic (Figure 14b), a dead leaves model (Figure 14c) and a Boolean model, (Figure 14d). This last model describes perfectly such structure (Quenec'h *et al.*, 1996; 1997). Indeed the Choquet capacity has been calculated on several compacts, (segment, hexagon, bi-point and triplet of points). It gives theoretical results in flawless agreement with experimental measures, obtained by morphological erosion with the same compact. The parameters of the model are the number of primary grains by volume and mean parameters of these grains. Moreover the theoretical connectivity number in R^2 coincides with experimental measures whatever is the time of sintering (figure 15). In addition one has been able to show that the activation energy and parameters of the kinetics of growth could be estimated correctly from the evolution of the parameters of the model.

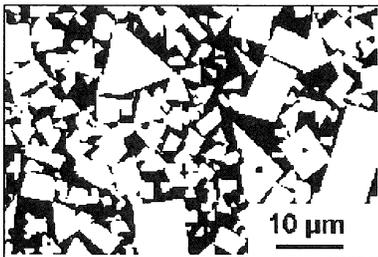


Figure 14a : Scanning electron microscope image of WC-Co cermet.

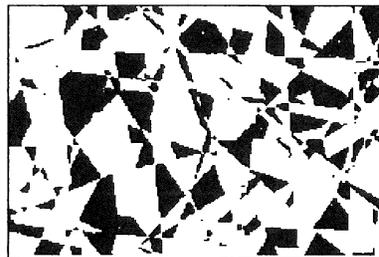


Figure 14b : Simulated two coloured Poisson mosaic.

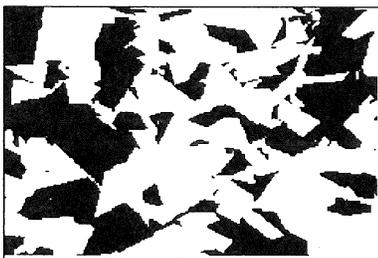


Figure 14c : Simulated two coloured dead leaves model with Poisson grains.



Figure 14d : Simulated Boolean model with Poisson grains.

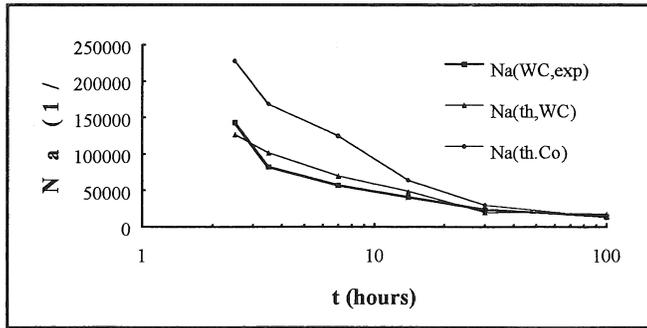


Fig. 15 : Connectivity number on the WC phase : experimental values, N_A (WC, exp) compared to values estimated while WC, N_A (th, WC) or Co, N_A (th, Co) are described by the Boolean model.

From the parameters of the Boolean model X of density of primary grains θ and Poissonian grain characteristic λ , it is also possible to estimate the number of connectivity in R^3 , $N_V(X)$ (Jeulin, 1979). So, this method allow to determine the fourth stereological parameter which may not be easily measured. $N_V(X)$ allows to obtain information about the topology of the structure in R^3 (Quenec'h et al 1997).

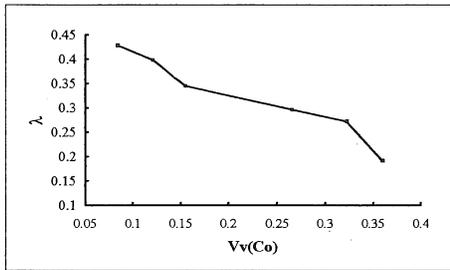


Fig. 16 : Variation of λ (in μm^{-1}) and Poisson grain characteristic λ , with the cobalt content.

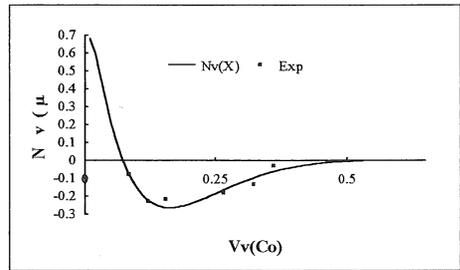


Fig. 17 : Variation of $N_V(X)$ (in μm^{-3}), number of connectivity in R^3 , with the cobalt content.

The values of $N_V(X)$ obtained are between -2.10^8 mm^{-3} for sample annealed 1 hour to -1.10^7 mm^{-3} (100 hours). These results put in evidence a very strong interconnection of WC grains which is in agreement with the presence of the WC skeleton observed on micrographs.

At last, for several samples sintered in the same conditions (furnace, temperature and duration similar) but with variable percentage of cobalt, it was then possible to estimate $N_V(X)$. As a first approximation, it was noticed that the parameter λ follows a linear law with the cobalt content (figure 16). The value of $N_V(X)$, in given conditions of sintering, is then available as a function of $Vv(\text{Co})$, the cobalt content (figure 17).

CONCLUSION

Several types of results regarding the quantification of the morphology of monolithic ceramics and cermets have been presented in this paper. It shows that automatic methods can be easily used for such investigations.

One of the most difficult step to solve is to obtain a correct image of the ceramic microstructure as the etching technique is not always easy to control without a bias (grain boundary missing, too deep (and wide!) grain boundaries,...). Contrarily to the biologists, the metallurgists and more specifically the ceramists have not to their disposal the many different types of stains to reveal microstructures. Nevertheless we have shown, in the case of rounded grains and in the case of WC-Co, that it is possible to use several types of segmentation to obtain automatically a correct result.

The $P(\ell)$ function is very useful and easy to perform. It gives access to many very important morphological parameters, which allow, for example to follow correctly different steps of sintering. Automatic image methods allows also to investigate the anisotropy and the homogeneity of materials.

Finally we have briefly shown also the power of probabilistic models to access to three-dimensional parameters and to simulate such ceramic microstructures (WC-Co, MOX, UO_2).

All these methods can be also used to quantify the morphology of any type of composite materials (see for example Boitier *et al.*, 1997a), or the damage in ceramics or civil engineering materials (Boitier *et al.*, 1997b; Redon *et al.*, 1997) in order to select correct hypothesis for mechanical modelling.

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REFERENCES

- Beucher S, Meyer F. The morphological approach to segmentation : the watershed transformation. *Mathematical Morphology in Image Processing* Dougherty ER ed. Marcel Dekker Inc 1992; **chap 12**: 433-481.
- Boitier G, Chermant JL, Chermant L, Doreau F, Vicens J. Morphologie par analyse d'images de composites à fibres longues : principes et résultats. *Rev Met CIT Sci Génie Mat.* 1997a; **94**: 1517-1536.
- Boitier G, Chermant L, Chermant JL. Morphological characterization of carbon silicon carbide composites. *Acta Stereol* 1997b; **16**: 275-280.
- Carle V. Ceramography of high-performance ceramics – description of materials, preparation, etching techniques and description of microstructures. *Pract Met* 1991; **28**: 359-377.
- Charollais F. Analyse d'images : un outil pour caractériser et modéliser la microstructure du combustible MOX. Thèse de Doctorat of the University of Caen, France, 1997.
- Charollais F, Bauer M, Coster M, Jeulin D, Trotabas M. Modelling the structure of a nuclear ceramic obtained by solid phase sintering. *Acta Stereol* 1997; **16**: 315-321.
- Coster M, Chermant JL. Précis d'Analyse d'Images. Les Editions du CNRS, Paris, 1985. 2nd Ed. Les Presses du CNRS, Paris, 1989.

- Coster M, Prod'homme M, Chermant L, Chermant JL. Morphological study during a ceramic process. *Microsc Microanal Microstruct* 1996; 7: 407-413.
- Coster M, Chermant JL, Prod'homme M. Quantification of ceramic microstructures. Q-MAT'97 Proceedings. Int^l Conference on the Quantitative Description of Materials Microstructure, Warsaw, Poland, 17-19 April 1997, Fotobit ed 1997: pp 115-124.
- Duval-Rivière ML, Vicens J, Carry C, Chermant L, Coster M. Deformed ceramics investigated by anisotropy parameters. STERMAT'94, International Conference on Stereology and Image Analysis in Materials Science, Wisła, Poland, 3-6 Oct 1994. Proceedings Ed Wojnar L. 1995 : pp 345-350.
- Exner HE, Hougardy HP. Quantitative Image Analysis of Microstructures, DGM Informationsgesellschaft Verlag, Oberursel, Germany, 1988.
- Gauthier G, Coster M, Chermant L, Chermant JL. Morphological segmentation of cutting tools. *Microsc Microanal Microstruct* 1996; 7: 339-344.
- Haas A, Matheron G, Serra J. Morphologie mathématique et granulométrie en place. *Ann Mines* 1967; 11: 736-753 and 12: 768-782.
- Hubner G, Haussner H. Material-orientated preparation of sintered ceramic bodies. *Pract Met* 1983; 20: 289-296.
- Jeulin D. *Advances in Theory and Applications of Random Sets*. World Scientific. 1997.
- Jeulin D, Villalobos IT, Dubus A. Morphological analysis of UO₂ powder using a dead leaves model. *Microsc Microanal Microstruct* 1995; 6: 321-384.
- Kurdy MB, Jeulin D. Directionnal mathematical morphology operations. *Acta Stereol* 1989; 8/2: 473-480.
- Lantuéjoul C. On the estimation of mean values in individual analysis of particles. *Microscopica Acta* 1980; 5: 266-273.
- Petzow G. *Metallographic Etching*. American Society for Metals, Ohio, USA, 1979.
- Quenec'h JL, Chermant JL, Coster M, Jeulin D. Example of application of probabilistic models : determination of kinetics parameters during liquid phase sintering. *Microsc Microanal Microstruct* 1996; 7: 573-580.
- Quenec'h JL, Jeulin D, Coster M, Chermant JL. Approach of liquid phase sintering process by probabilistic models. *Advances in Theory and Applications of Random Sets*. Ed Jeulin D. Fontainebleau 9-11 October 1996. World Scientific Company. 1997: pp 231-249.
- Redon C, Chermant L, Chermant JL, Coster M. A mechanical damage model based on the measurement of microcrack orientation in concrete by Fourier Transform. *Acta Stereol* 1997; 16: 287-292.
- Rhines FN. *Microstructology*. *Pract Met* 1986; 23: 1-11.
- Serra J. *Image Analysis and Mathematical Morphology*. Academic Press. New York, 1982.
- Stoyan D, Schnabel HD. Description of relations between spatial variability of microstructure and mechanical strength of alumina ceramics. *Cer Int* 1990; 16: 11-18.
- Virkar AV, Gordon AS. Fracture properties of polycrystalline lithia-stabilized β -alumina. *J Am Cer Soc* 1977; 60: 58-61.
- Zacharie I. Traitements thermiques de l'oxyde d'uranium irradié en réacteur à eau pressurisée: gonflement et relâchement des gaz de fission. Thèse de Doctorat of the Ecole Centrale de Paris, France, 1997.