MORPHOLOGICAL CHARACTERIZATION OF SiC MATERIALS

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Résumé - L'analyse quantitative d'images a été utilisée pour mesurer les principales caractéristiques morphologiques de quelques nuances de SiC-Al : caractéristiques granulométriques, forme et anisotropie des cristaux. L'homogénéité de ces céramiques a été étudiée par des méthodes statistiques.

Abstract - Quantitative image analysis were used to measure the main morphological characteristics of some SiC-Al materials : size distribution characteristics, shape and anisotropy of the SiC-crystals. Statistical methods were utilized to inform on the homogeneity of these materials.

I - INTRODUCTION

For ceramic materials the role of the microstructure on the mechanical or electrical properties is very important. For example, the creep behaviour of SiC materials is governed by the amount of the intergranular phase, its distribution between the boundaries, and also by the mean grain size of the SiC crystals /1,2,3/. In these conditions it is necessary to define the morphology of the microstructure of ceramic materials and quantitative image analysis is strongly advised /4/.

The aim of this paper is to show what types of parameters are determined using this technic in the case of silicon carbide materials. The first problem to solve is to obtain a correct image. This requires a perfect metallography.

II - TECHNICAL PROCEDURES

Four batches (from Elektroschmelzwerk, F.R.G.) of SiC with aluminium additive were used: SiC-AO3 with 0.3 wt % of Al and SiC-Al5 with 1.5 wt % of Al. This last batch has been heat treated during two different times and noted SiC-Al5 E1 and E2, /1/. These materials have been polished using classical methods : diamond pastes of different mean grain sizes (14, 7, 3 and 1 μm) and then a chemical etching in a boiling Murakami solution (24 g NaOH + 42 g $\text{FeK}_3(\text{CN})_6$ in 100 cm^3 H_2O), during 5 mn. Optical micrographs of SiC-Al 1.5 wt % are shown on figure 1 for two mean grain sizes.

Fig. 1 - Micrographs of SiC-Al5 and SiC-Al5 E2.

These structures are typical examples of a mosaic structure. Regarding the morphological characteristics of such materials we have to investigate the mean grain size characteristics and their shape. As these materials are obtained by hot-pressing technic, a certain anisotropy in the crystals can arise. Finally we have also to approach the homogeneity of these materials by using the statistical methods. This work has been performed with a texture analyzer system, TAS, from Leitz, which uses an hexagonal grid. Its design is based on mathematical morphology which allows to improve the images /5/.

Measurements have been carried out on micrographs. After detection and memorization it was necessary to thin the grain boundaries as they shift the results when they reach a certain thickness. To obtain the minimum thickness, of one pixel, skeletonization by influence zone of the crystals has been utilized /6/. This skeleton was obtained by using the L_6 neighbourhood configuration followed by a clipping by the E_6 neighbourhood configuration. Then the image is ready for measurement (Fig. 2).

Fig. 2 - Skeletonized image superposed to its optical micrograph and detected image with the crystals totally included in the frame of measurement.

III - GRAIN SIZE CHARACTERISTICS

Among the different possible grain size methods, we have chosen the analysis particle by particle as these structures do not present interconnected phases and as the crystals are sensitively convex. The crystal surface area, A_i , totally included in the frame of measurements has been measured. To take into account that each grain does not possess the same probability to be included in this frame of measurements, a corrective method on the numbering, called frame measurement correction /7/, has been used : to each particle totally included in the frame of measurement, one assigns a statistical weight, equal to the ratio of the surface area of the frame of measurements over the surface area of this same frame, but eroded by the minimal rectangle circumscribed to the particle.

On figure 3 are presented the results in the mean grain size density form. From these curves, the interquantile deviation between the values of the measured surface areas for a frequency of 25, 50 and 75 %, has been calculated for each batch of SiC. In order to compare these distributions to a normal logarithmic law, Henry tests have also been plotted (Fig. 4). These different results are listed on Table I.

From this table one notes that :

- the mean grain sizes of the SiC crystals for SiC-A03 and A15 are sensitively of the same order of magnitude,
- after thermal treatment of the SiC-A15 batch, the crystal growth of SiC-A15 E2 batch is very important. This growth does not modify sensitively the grain size distribution,
- the analysis of the reduced interquantile deviation shows that a stability in the grain size dispersion exists. The same result is indicated, with less accuracy, by the values of the standard deviation. This last one is, in fact, very affected by the extremities of the distribution which are depending on much more important errors,
- regarding the shape of the distribution curves, gausso-logarithmic scale has been

Fig. 3 - Mean grain size density distribution for the four batches of SiC.

Fig. 4 - Henry test for the four batches.

used to plot the cumulated frequencies. One notes that the grain size distributions of materials without growth follow a logarithmic-normal law. On the other hand, those for materials with growth (E1 and E2) deviate notably from this law. This is probably due to the phenomena of accommodation of grains and of the anisotropy of crystalline growth.

Characteristics	SiC A03	SiC A15	SiC A15 E1	SiC A15-E2
Mean area $A(\mu\text{m}^2)$	3.0	3.2	5.5	9.3
Standard deviation $\sigma(\mu\text{m}^2)$	3.6	3.0	4.6	10.3
Reduced standard deviation $\sigma/\bar{A}$	1.20	0.94	0.84	1.11
$A\ 1/4\ (\mu\text{m}^2)$	0.8	1.0	1.9	2.2
$A\ 1/2\ (\mu\text{m}^2)$	1.5	2.0	3.0	4.9
$A\ 3/4\ (\mu\text{m}^2)$	3.3	4.0	6.2	10.3
$A\ 1/2 - A\ 1/4\ (\mu\text{m}^2)$	0.7	1.0	1.1	2.7
$A\ 3/4 - A\ 1/4\ (\mu\text{m}^2)$	2.5	3.0	4.3	8.0
$A\ 3/4 - A\ 1/2\ (\mu\text{m}^2)$	1.8	2.0	3.2	5.4
Reduced interquantile deviation ($A\ 3/4 - A\ 1/4$)/ σ	0.83	0.94	0.78	0.86

TABLE I
Grain size characteristics of the SiC batches.

IV - SHAPE

As the crystals are not always equiaxed, we have characterized their shape by using several shape indexes. Among the numerous possible parameters, we have chosen three shape indexes [4] :

- the most general, noted $F1 = 4\pi A/(L_2)^2$, where A and L_2 represent respectively the mean surface area and the mean perimeter of the crystals,
- $F2$, which represents the deviation from the convexity. It is measured by the ratio of the convex hull perimeter over the object perimeter,
- $F3$, which characterizes more specially the anisotropy : each grain is modeled by an ellipse, the eccentricity of which is obtained from the measurement of the projections in the three directions of the hexagonal grid.

Results are given in Table II. From these, one notes that :

- $F1$ does not appreciably evolve,
- the deviation from the convexity is extremely small : the crystals remain there-

	SiC AO3	SiC Al5	SiC Al5-E1	SiC Al5-E2
F1	0.68	0.69	0.71	0.68
F2	0.98	0.99	0.99	0.99
F3	0.72	0.72	0.73	0.79

Table II
Values of the shape
indexes used.

- fore always convex whatever the batch is,
- the values of parameters F3, deviation from the eccentricity, are approximately of the same order of magnitude for SiC-AO3, Al5 and Al5-E1 materials. These values point out that the crystals are perceptibly elongated. For SiC-Al5 E2 the value of this shape index is much higher. This indicates that this material has suffered a non-equiaxial growth. The fact that the crystals are individually anisotropic does not mean that the material is globally anisotropic. This necessitates a more important investigation of this characteristic.

V - ANISOTROPY

To characterize the anisotropy of relatively convex grains, either the intercept rose [8] or the direction rose [4,5] can be plotted. As the rose of direction is more sensitive to the anisotropy, we have chosen this last method.

By definition, the rose of directions is the length of the grain boundaries oriented in a given direction, divided by the surface area of the frame of measurements (Fig. 5). With the hexagonal grid, these lengths have been measured according to 6 orientations: 0 , $\pi/3$, $2\pi/3$, $\pi/6$, $\pi/2$ and $5\pi/6$, using a neighbourhood transformation belonging to each direction.

Fig. 6 - Roses of directions for SiC-Al5 and Al5-E2.

Fig. 5 - Schematic of the measurements with the rose of directions.

Results are reported on figure 6. One notes that the metallographic sections, for which the main direction is parallel to the compression axis, show an anisotropy more appreciable than that observed for the perpendicular direction. This anisotropy can also be modeled with an ellipse for which the axis have been calculated graphically (Table III). The eccentricity, e , can then be calculated from the relation :

$$e = \sqrt{1 - \left(\frac{a}{b}\right)^2}$$

where a and b are the values of the half-axis of the ellipse.

In all cases, a small effect of the anisotropy is observed in the analysis direction parallel to the compression direction. This anisotropy effect is especially important since the material has been subjected to a large thermal treatment.

analysis direction	SiC Al5		SiC Al5-E2	
	$\perp$	//	$\perp$	//
a/b	0.84	0.69	0.76	0.53
e	0.54	0.72	0.64	0.85

TABLE III
Values of eccentricity for
the batches SiC Al5 and
Al5-E2.

VI - MORPHOLOGICAL HOMOGENEITY

Using sintering processes, materials are generally very homogeneous compared to materials obtained by usual metallurgy. So in a first attempt, a classical analysis can be sufficient to precise this homogeneity.

On each analysis frame, we have measured the connexity number in R^1 (number of grains per unit length) and in R^2 (number of grains per unit surface) in order to test the homogeneity of these materials. The implied model assumes that the micro-structure is uniform and that the setting of the frame is such that each variable is independant from one frame of measurement to the other. A regular setting was chosen in taking a net large enough with regard to the structure investigated. On Table IV are given the results of SiC-Al5 E2 in two perpendicular directions. The Student test on this sampling shows that these materials present a very large homogeneity : in the most unfavourable case, we have a 93 to 96 % probability to find the mean value in an interval of 2.5 % for N_L and 97 to 98 % in an interval of 5 % for N_A .

	N_L	$\sigma(N_L)$	$\Delta N_L (2,5\%)$	P	$\bar{N}_A$	$\sigma(N_A)$	$\Delta N_A (5\%)$	P
direction 1	838	40.72	20.95	0.96	128307	10503	6415.35	0.98
direction 2	675	38.37	16.87	0.93	126216	11867	6310.80	0.97

TABLE IV
Results concerning the sampling of the batch SiC Al5-E2.

Results relative to N_A are a somewhat less good. This is normal because N_A is obtained from the second derivative while N_L is obtained from the derivative.

VII - CONCLUSION

Such materials are now well characterized from the morphological point of view. It is then possible to have experimental microstructural values which can be used to explain deviations from well defined phenomenological relationships.

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