Microstructural characterization of TiO₂ ceramics substrates using image analysis software ImageJ

C. de S. Pereira¹, E. E. de M. Oliveira¹, T. G. de Paula².

E-mail: <u>cristina@ien.gov.br</u>, <u>eemo@ien.gov.br</u>, <u>thaisgonçalvesdepaula@eq.ufrj.br</u>

¹ Serviço de Tecnologia de Materiais e Química, Instituto de Engenharia Nuclear - CNEN/RJ. ² Escola de Química, - UFRJ.

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In this work the clusters size and porosity of TiO₂ ceramics substrates were quantified. The ceramics analyzed were prepared by two techniques: slipcasting and compaction, starting from an aqueous suspension of commercially available TiO2 (Rutile RKB-2, B'Herzog) with organics additives. The optimum sintering temperatures to obtain support with good mechanical resistance and adequate porosity to be applied as a ceramic substrate was 1100 and 1150°C. The microstructure of the ceramics and the clusters of the atomized suspensions in the spray dryer were analyzed in scanning electron microscopy (SEM-HITACHI TM 3000). The image analysis software ImageJ was used to treat images to removing noise and highlight the regions of interest in the analysis. The porosity was determined from analysis of five different images using the following algorithm, as suggested in the standard ASTM E 1382-97 [1]: Unsharp Masks Filter; Median Filter; Threshold Command. The average clusters size was determined using Ferret Diameter [2], from analysis of five different images, reaching the count of 1126 clusters. It was used same algorithm above, just applying at the end, the Overlay/Masks command to highlight the clusters contour. The porosity results obtained by ImageJ were compared with values obtained using a method based on the Geometric and Hydrostatic porosity [3], which showed good agreement, as shown in the Table 1.

Table 1- Comparing of porosity results of TiO_2 ceramic obtained by digital image analysis (ImageJ), geometric and hydrostatic porosity methods.

Molding	Geometric	Hydrostatic	ImageJ
Compaction	34.83 ± 4.52	34.60±5,17	32.97±3.38
Slip casting	32.38±3.15	32.55±1.41	34.74 ± 2.34

The Figure 1 shows the image treatment steps of TiO_2 ceramic substrate, while the Figure 2 shows image processing step to measure clusters size.



Figure 1- Step of image analysis of TiO_2 ceramic by software ImageJ: Original image (1A), after applying filters (1B) and image binary (1C).

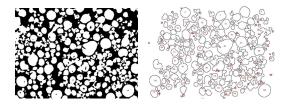


Figure 2- Determination of the clusters size of atomized TiO_2 suspension in spray dryer.

Applying methodology described for determination the average clusters size, the value of 7.174 μ m \pm 0.984 was obtained (Figure 2). The results showed that the digital image processing was effective in the analysis of microstructure of ceramic substrates, as well as, for determination of particle size in micrometric scale. The porosity determined by images analysis considers in addition to the open pores, also the closed pores. The next stage will be the microstructural characterization after polishing and chemical attack of the sintered substrate surface for the analysis of grain size and porosity.

References

- ASTM STANDARD E1382-97, Standard test methods for determining average grain size using semiautomatic and automatic image analysis. American Society for Testing Materials, West Conshohocken, EUA, 2004.
- [2] MARCOMIN R.; SOUZA D. M. F. Caracterização microestrutural de materiais cerâmicos utilizando o programa de processamento digital de imagens ImageJ, Ceramic, 57 (2011) 100-105.
- [3] ASTM MANUAL OF WATER AND ENVIRONMENT TECHNOLOGY C373-18, Standard test methods for determination of water absorption and associated properties by vacuum method for press ceramic tiles and glass tiles and tiles boil method for extruded ceramic tiles and non-tile fired ceramic whiteware products. American Society for Testing Materials, NY, 2018.