Short communication

Computer controlled digital microscopy—Wide area characterization of waste incorporated clay ceramics

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\begin{abstract}
A computer-controlled digital microscope coupled with a motorized optical system was used to analyze wide surface areas of a clay ceramics incorporated with 10% waste from paper recycling. This system acquires perfectly matched mosaic images covering the sample full surface and allows image analysis to interpret size, shape and spatial distribution of incorporated phases. In bright field, quartz particles, naturally existing in the clay, impair the paper waste observation. In dark field, the contrast disappearance of the quartz allows the paper waste to be analyzed.
\end{abstract}

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1. Introduction

Reported results on clay incorporated with a waste in the form of sludge from paper recycling treatment, provided evidence of significant changes in the ceramic structure and properties [1]. Scanning electron microscopy (SEM) directly revealed porosity and quartz particles. By contrast, the waste particles could only be indirectly detected by energy dispersive spectroscopy (EDS). Consequently, a large scale quantitative analysis could not be performed due to inherent limitations of the SEM/EDS technique. In this note, the microstructure of a similar clay ceramic, either plain or incorporated with paper waste, was analyzed by two steps, bright and light fields, computer-controlled digital optical microscopic method. Despite the several existing methods using pattern recognition software coupled to optical systems, the present two step one permitted not only the complete microstructure characterization of distinct dispersed phases in a wide cross section area of the ceramic sample but also its separate quantitative evaluation.

2. Experimental procedure

A clayey body composed of kaolinite clays mixed with quartz sand, typically used in the fabrication of conventional bricks and tiles in Campos dos Goytacazes, state of Rio de Janeiro (RJ),
Brazil, was incorporated with paper waste obtained from the primary treatment stage of a recycling plant in Santo Antônio de Pádua, RJ. The fabrication of the 10 wt.% waste incorporated as well as the plain clay ceramics was described elsewhere [1]. In short, 11 mm thick rectangular bodies (114 mm × 25 mm) were press-molded at room temperature and stove-dried at 110 °C. Final samples were obtained by firing at 750 °C for 2 h, which was enough to consolidate the ceramic structure [2]. The largest surface of the sample was polished and analyzed by both dark field (DF) and bright field (BF) optical microscopy. A computer-controlled model Axioplan 2ie Zeiss optical microscope acquired digital images by means of a Zeiss AxioCam HR camera. The whole system was commanded by the Axio Vision software, which also provided image analysis functions for the quantification of phases. A magnification of 50× and a resolution of 2.11 μm/pixel were attained. At this magnification, each field image covered 2.73 mm × 2.17 mm. Since the paper waste particles could eventually surpass the image size, mosaics were assembled to cover full phase dimensions in the sample. Perfectly matched images were obtained as described elsewhere [3].

3. Results and discussion

Fig. 1 shows a mosaic assembly for the neat clay (0% waste) composed of 9 × 5 = 45 individual images and a total area of 24.7 mm × 10.9 mm. The images were acquired in BF mode. The bright spots in this figure are actually quartz particles as they reflect light in a specular mode. An automatic quantitative analysis of this mosaic revealed a 14.7 vol% uniform distribution of quartz particles throughout the section.

Fig. 2 shows similar mosaic assembly for the 10 wt.% waste sample. Even though the paper waste particles are visible in this figure, as the larger gray regions, their contrast is not optimized. Segmentation of this kind of image for automatic quantitative analysis is hard to perform owing to the multimodal aspect of its histogram [4]. To bypass this shortcoming, mosaic assemblies of both samples, neat and 10 wt.% incorporated were also acquired in DF mode, as shown in Figs. 3 and 4, respectively. In these figures, it is important to note that all specular reflecting quartz particles are now hardly visible. By contrast, the diffuse light scattered from the paper waste particles, Fig. 4, is clearly imaged. Actually, there is a slight loss of sharpness in Figs. 3 and 4, which is a characteristic of the DF mode of image formation. However, this is not relevant in the quantification of size, shape and spatial distribution of paper waste particles, as most of them are very large at this magnification.

The loss of sharpness in Fig. 4 can be partially compensated through image processing. Thus, a standard routine was developed to analyze second phase particles. This routine followed the typical steps of pre-processing, segmentation, post-processing, and measurement, as previously described [4]. In the pre-processing step, an edge enhancement filter is used to improve the sharpness of particle edges. In the segmentation step, the particles are automatically discriminated by their brightness employing a computerized method based on the Otsu algorithm [5]. Small bright artifacts and holes in Fig. 4 were automatically eliminated in the post-processing step. Fig. 5 shows the resulting processed image of Fig. 4 obtained by means of this automatic routine, which permits to evaluate several stereological parameters. As an example, the volume fraction of the paper waste particles was measured for the full section in Fig. 5 and found to be 11.4 ± 2.0%. By subdividing Fig. 5 in three equal parts along the horizontal direction, the corresponding volume fractions were somehow different. The left part was found to have 9.8 ± 1.3%, the middle, 13.3 ± 0.9%, and the right part 11.3 ± 1.1%. These results attest the discrimination power of the technique in
characterizing non-uniform distribution of incorporated particles to a ceramic matrix.

4. Summary

A digital motorized and computer-controlled microscope technique permitted a wide section of a paper waste incorporated clay ceramic microstructure to be analyzed. Using both bright and dark field mosaic-assembled images, it was possible to identify and discriminate quartz and paper waste particles. A segmentation method based on the Otsu algorithm allowed a precise identification of the paper waste particles and the evaluation of their spatial distribution. This enables the detection of non-uniform distributed particles, within the statistical dispersion, in the wide section analyzed.

Conflicts of interest

The authors declare no conflicts of interest.

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REFERENCES