ON-LINE MEASUREMENT OF CERAMIC POWDER GRANULOMETRY BY COMPUTER-AIDED IMAGE ANALYSIS

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ABSTRACT

In this paper the development of a ceramic particle size measurement technique based on Computer-Aided Image Analysis (CAIA) has been developed, which should be much quicker than image analysis by microscopy, much more accurate than industrial sieving and less costly with respect to laser diffraction. Another main advantage of this technique is the possibility of on-line particle size control at the exit of the spray dryer, for quick feedback to the spray-drying process. The uncertainty of the measurement technique is discussed and laboratory and on-line results are reported.

1. INTRODUCTION

The particle size distribution of the powder after the slip spray-drying process is a parameter of fundamental importance in order to have a good structural behaviour of both green and fired ceramic tiles. Particle distribution should be kept within precise limits, in such a way as to have optimal and uniform characteristics in the final product.

There are several spray-drying parameters that influence the granulometry of the powder, in particular:

- quantity of water and viscosity of the slip, which determine how the slip will be atomised through the nozzles;
- pump pressure, which influences the behaviour (e.g. atomisation, trajectory, etc.) of the particles coming out of the nozzles;
- geometrical characteristics and position of the nozzles, which directly influence the particle size distribution.

In order to have a greater, faster control of the particle size distribution, usually more variables are regulated at the same time. It is clear that different types of raw materials will be spray dried with different results. Therefore a specific process set up should be used for each material.

Nowadays, in the ceramic industries, particle size distribution is usually controlled off-line using electro-mechanical sieving devices [Venturi, 1992]. The samples are taken from the line at intervals of about 3-4 hours, but this kind of control does not allow quick feedback for spray-drying process control. An on-line, non-intrusive measurement technique is thus desirable, since it could allow continuously regulating the spray-drying parameters, in such a way as to keep the powder product within the desired limits.

The choice of the measurement technique is important for several reasons. First of all, different methods could give different measured quantities (e.g. volume, weight, surface or number) and consequently different particle size distributions, which can not be easily compared without introducing the corresponding uncertainty factors. For example, in order to convert the data from percent distributions in counts (usually measured by the new instruments) to weight, it is necessary to assume the particles have spherical shape and constant specific weight. However, it is well known that this is not true in the case of ceramic powder. This point is in practice very important, since all the data generally employed in last few years as references in the industry are expressed in weight distributions, as they have been measured by sieving methods. In order to minimize these errors, it also appears important to know the particle shape.

However, in dealing with industrial applications, accuracy, velocity and low cost are all requirements that a sensor must have at the same time. A compromise should be thus found, in order to integrate these properties in an optimal way.

This point of view has been followed in the present work. After reviewing the state of art for the particle size measurement techniques (Par. 2), a new approach has been proposed, with the aim of developing an on-line non-invasive measurement technique with an accuracy comparable to that of the most recent and sophisticated sensors (mainly based on laser diffraction), but with lower costs. A technique based on image acquisition by high-resolution CCD cameras and processing has been chosen for this application. In the paper, the measurement chain (Par. 3) and some experimental results (Par. 4) are presented and the uncertainty analysis is discussed (Par. 5).

2. METHODS FOR PARTICLE SIZE MEASUREMENT

Spray-dried ceramic particles generally have a toroidal shape with an equivalent diameter ranging from 90 to 600 μm. Their frequency distribution is similar to that of a heterogeneous sample, with the maximum usually found around the smallest values. This feature leads to dimensional segregation of the sample, in the sense that the particles will not have a random spatial distribution, but distribution will be influenced by particle dimension. This makes sampling of the powder from the line difficult: ceramic particles are often fed into hoppers or transported on belt conveyors, hence the feeding itself can generate both vertical and horizontal segregation. For these reasons, it is recommended that, wherever possible, the "golden rules" of sampling should be followed, [Allen, 1997]:

- A powder should always be sampled when in motion.
- The whole of the stream of powder should be taken for many short increments of time in preference to part of stream being taken for the whole of the time.

Observance of these rules, coupled with an understanding of the manner in which segregation takes place, leads to the best sampling procedures.

In the ceramic tile industry the powder is transported on belts. Sampling must be performed directly on belt in whole free flow in a short period of time. Some indications concerning the proper techniques for sampling and the methods for granulometry distribution measurement can be found in the literature [Arai, 1996].

The following measurement methods are suitable for ceramic powder:

SIEVING METHOD:

The sieving method is the oldest and still the most widespread one [Arai, 1996]. A number of sieves with openings of different sizes are arranged in order in a vertical stack with the sieve having the largest apertures at the top. After a 100g of powder sample has been charged to the top of the sieve, a vibrator agitates the sieve stack for about 5 minutes. The particles larger than the size of each opening are retained on the upper side of each sieve. The size distribution of the powder sample is easily determined by weighing the particles retained on each sieve. This method is very convenient for its simplicity and low cost. However, it is subject to operator errors in all the measurement phases. Its measurement precision is comparatively large owing to adhesion to the sieve or cohesion between particles. Recently, an interesting new version of this method has been proposed [Sonic Sieving Accurately Measures Fine Particle Size Ceramic Industry], called "sonic sieving", in which the sieves are not vibrated, but only the powder is put in motion using acoustic waves produced by a low-frequency loudspeaker.

LIGHT SCATTERING OR DIFFRACTION METHOD:

A simple light-scattering photometer measures the angular distribution of the intensity of polarized light scattered by a particle sample [Hulst, 1981]. The light intensity scattered by particles less than 0.1 μm in size is proportional to the 6^{th} power of the diameter. For larger particles, up to approximately 4 μm , the scattering light intensity is proportional to the square of their diameter for most scattering angles. The light

scattering system defines a rectangular measurement volume by imaging rectangular apertures with the sending and receiving optics. A particle passes through the illuminated measurement volume and scatters light that is received by the detector optics and focused onto a photodetector. Each optical signal is converted into an electrical pulse that is processed electronically. The signal processor counts these pulses and builds up a particle size histogram. According to the light obscuration method, when a particle passes through the collimated beam of light, a shadow is cast on the light detector that is proportional to the particle size. In practice, the particle "eclipses" the light reaching the detector. The light detector responds to the maximum cross sectional area presented to the light and measures the particle equivalent diameter [Heuer & Leschonski, 1985]. These sensors present the advantages of having high accuracy and quick responses, but they are expensive, thus limiting use mostly to laboratories.

ACOUSTIC SPECTROSCOPY PRINCIPLE:

Sound waves interact with objects in a similar manner to light, but have the advantage of being able to travel through concentrated suspensions and emulsions [Hackley, 1996]. It uses ultrasound frequencies, typically from 1 to 200 MHz. The attenuation of these waves is then accurately measured. The measured attenuation of sound waves as a function of frequency is known as the spectral data. There is a direct correspondence between the particle size distribution/concentration and the associated spectrum. At present, the acoustic spectroscopy sensors are at a development stage, which is suitable only for laboratory applications.

3. THE PROPOSED MEASUREMENT TECHNIQUE

The proposed measurement technique is based on Computer-Aided Image Analysis (CAIA) and basically measures the area of the particles present in a recorded scene. This method performs a frequency distribution in counts, as it examines the particles one by one.

With regard to the particle size to be measured, some statistical diameters are presented in the literature [Arai, 1996]. In this paper, the characteristic dimension of a given particle is the Heywood diameter (diameter of the circle having the same projected-surface area of the particle). This diameter is statistically acceptable, and is preferred to other available parameters (e.g. Feret or Martin diameters), because it takes into account both dimensions of the object being measured.

The measurement system is composed of two steps: image acquisition and image processing.

The measurement chain is schematically shown in Figure 1. The experimental set-up was first developed in the lab and then tested in line. The powder is positioned on a metallic plate with a colour giving a high contrast for the particles. Then, the plate is excited in vibration at its first resonance frequency using a loudspeaker, in such a way as to separate and disperse the particles within the samples, until each particle is clearly individuated on the plate.

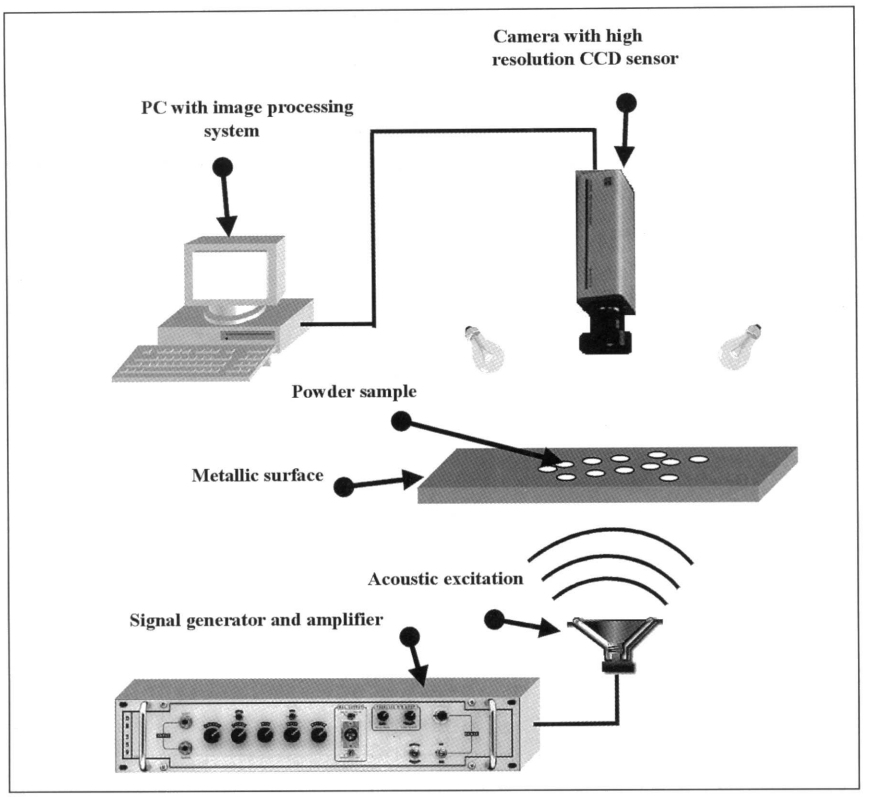

Figure 1. *Measurement chain based on image acquisition and processing.*

At this point the scene is acquired by a black and white, high resolution CCD camera with a 60 mm macro objective, whose main technical specifications are shown in Table 1.

Type CCD	Progressive scan interline
Number of elements	1008 x 1018
Colour	Black and white
Pixel pitch	9 x 9 μm
Array of micro lens	Standard, fill factor 60%
Output digital	Parallel 8 bit
Active area	9.1 x 9.2 mm
Frequency of maximum acquisition	30 Hz
Range of temperature	0 ÷ 40°C.

Table 1. *Main technical specifications of CCD camera*

Once the image is acquired, it is processed by specifically designed software developed in the LabView environment. This set-up is important in order to have the measurement and the data processing steps separated, in such a way as to improve flexibility. The measurement range only depends on the magnification factor of the optics used, while the same software can be utilized for every range of interest. Furthermore, starting from a single image, it is possible to extract other important information concerning the analyzed sample, simply by modifying the processing routines. For example, parameters relating to the morphology of the particles could be extracted, as well as other types of characteristic diameters.

Finally, it should be considered that, thanks to the recent decrease in the costs of CCD sensors, the system is significantly cheaper than laser diffraction sensors, thought it has comparable time response and measurement accuracy, as shown in the following paragraphs.

3.1 THE MEASUREMENT CHAIN AND ITS RESOLUTION

The resolution of an image indicates the real dimension of each pixel on the image. In practice, the portion (and thus the dimension) of the scene effectively acquired is determined by the projection of the sensor, through the optics, on the scene. In this situation, the illumination condition also plays an important role and should be thus carefully considered.

A scene illuminated in a traditional way may lead to two problems on the image:

1. The shadow of each particle may cover the image of the surrounding particles. This problem can be minimized by having an illumination as orthogonal as possible to the plate, in such a way as to highlight only the maximum section of the particle, without interfering with the other ones.
2. The intensity of the light diffused by the particle toward the camera tends to zero when the observed point is on the particle edge. This effect is not completely limitable, since a transition zone between particle and background will always be present.

In practice, the illumination condition should be optimized by trying to minimize the interfering inputs due to the two problems. From experimental tests (not presented in this research), it was shown that using a traditional illumination set-up (as shown in Figure 1), the two effects are negligible for the present application. It is important to have the lamps symmetrically disposed with respect to the scene of interest.

Once the viewing angle of the camera and the illumination condition are fixed, the resolution depends only on the distance between camera and scene. The numerical value of the resolution was found through a dedicated calibration, performed at the maximum allowable resolution, i.e. with the maximum lens magnification (1:1) and at the minimum distance. A calibre with known dimensions was employed as reference. The resolution achieved in this work is therefore equal to the dimension of the pixels, i.e. 9 μm /pixel.

3.2 DIGITAL IMAGE PROCESSING PROCEDURE

The image processing procedure is performed with the aim of extracting from the scene the information of interest and of compressing the amount of data [Weeks, 1996]. In the present application, the relevant parameters are the particle area, perimeter and number in the analysed scene. The image is composed of 1008x1008 pixels with 256 grey levels. Considering that the resolution is 9 μm/pixel, the dimension of the scene will be about 81 mm^2, which is also the dimension of the CCD sensor.

The acquired image must be conveniently processed before any measurement is done, because the particles must be highlighted with respect to the background. The acquired image is first thresholded, then separated and at last we proceed to count and measure the particles. The following steps are specifically performed:

De-interlacing filter: the effect due to interlacing phenomena could have significant influence on the measurement accuracy, since the edge of the particle may become too irregular (Figure 2). In order to reduce this effect, a series of filters was applied directly on the acquired image before the threshold application, in such a way as to minimize the filter-induced variation on the grey levels. The results are satisfying, as shown in Figure 2.

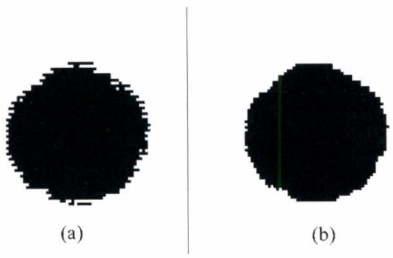

Figure 2. *Interlacing effects on a particle image (a); effects of the de- interlacing filter (b).*

Laplacian filter: it is used to enhance the contrast of the particle image with respect to the background, by highlighting the edges between dark and bright regions.

Threshold: an 8 bit grey scale acquired image is reduced in a binary one. The algorithm receives the threshold value as input, then sets to 0 all the pixels below the threshold value and to 1 the pixels above. The threshold value is critical to achieve good results. Its value is automatically chosen using the Clustering Technique [Jain, 1989]. This algorithm analyses the image histogram and calculates the centres of gravity of the major histogram regions, Figure 3. Then it computes the distance between each grey level and the centres of gravity and assigns the grey level to its nearest centre. In this way the grey levels (and hence the image itself) are divided into groups or classes (two in the present case), one representing the objects and the other the background. The automatic selection of the threshold value is very important for on line application.

Filter for hole filling: the diameter of Heywood is based on the measurement of the total particle area. Therefore, it is correctly computed only if the particle is filled, while ceramic particles have a hole in the center because of their toroidal shape. The applied filter is based on connectivity, i.e. on a criterion used to decide when two pixels belong to the same line. In the present case a degree of connectivity equal to 8 was employed (the pixels above-below, on the right, on the left and on the diagonals were considered).

Erosion: it is used to eliminate spurious and noisy pixels. This filter, according to the connectivity assigned to each object, detects the particle edge and eliminates a number of pixels equal to the number of performed erosions. In this work erosions with a degree of connectivity equal to 8 were performed. Using this value and having a resolution of 9 μm /pixel, particles with a diameter of maximum 18 μm are eliminated. This assumption seems correct, since this diameter is significantly below the usual range of interest (90μm -600μm) and the related particles can be thus assimilated to noise.

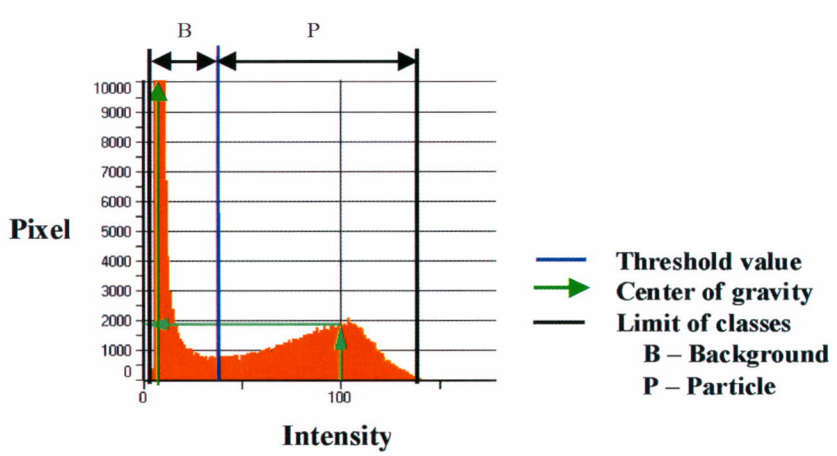

Figure 3. *Clustering Technique visualization for threshold computation.*

Separation: during the spray-drying process, two or more slip drops may be dried together, thus forming an agglomerate. This means that during the separation step the image processing software must be able to distinguish between agglomerates and adjacent particles. This recognition is performed according to the dimension of the connection between the particles.

The binary image is subjected to a specified number of erosions (a particular kernel which "erodes" the borders of objects, as previously stated). Then the objects are labelled according to the specified connectivity (parameter used to determine whether two pixels are adjacent or not) and finally the original image is reconstructed without the touching regions. This allows having clearly distinct objects without any significant loss of information compared with the original image. The final image differs from the original one only for the elimination of the smallest connections.

Elimination of the particles on the image edge: the image of the particles on the edge of the measurement area may not be completely acquired, introducing incomplete information. Therefore, these particles are eliminated.

Figure 4 shows the effects of the different applied filters.

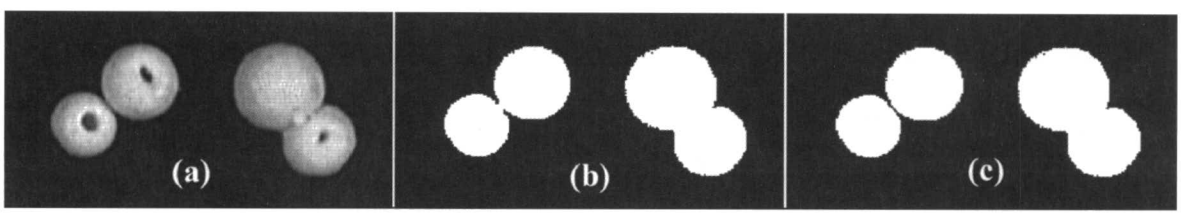

Figure 4. *Sequence of applied filters: (a) de-interlacing, (b) hole filling (c) separation.*

3.3 MEASUREMENT ON THE BINARY DIGITAL IMAGE

The measurement algorithm takes a pre-processed image in input and, according to the specified connectivity, the number of pixels belonging to each particle is simply counted. Given a resolution value, the software measures the area in pixel or in μm^2 and the perimeter in pixel or μm . The Heywood diameter can be computed from the value of the area. For each *m*-th acquired image (several images are acquired in different zones for each sample, in order to improve the statistics), having determined the total number of particle and the Heywood diameter for each one, the particle size distribution can be computed using Equation 1:

$$F_{m,i} = \frac{n_{m,i}}{\sum_{i=1}^{I} n_{m,i}} \qquad \text{[Eq. 1]}$$

where:

$F_{m,i}$: partial fraction of particles with Heywood diameter in the *i*-th interval of the distribution;

$n_{m,i}$: number of particle, measured in the *m*-th acquired image, with Heywood diameter in the *i*-th interval of the distribution;

I : total considered range of particle size.

If N partial acquisitions are performed, the final average value can be computed by Equation 2:

$$\bar{F}_{N,i} = \frac{1}{N} \cdot \sum_{m=1}^{N} F_{m,i} \qquad \text{[Eq. 2]}$$

The measure is stopped when the difference between two consecutive values of $F_{N,i}$ is below a specified threshold for all the intervals.

4. RESULTS

A typical measurement result is shown in Figure 5. The sample is composed of powder for porcelain tile production. In order to have an idea of the measurement repeatability two results taken on the same sample are compared. Maximum discrepancies are below 3-5 % for each interval, confirming therefore the satisfactory performance of the proposed technique.

Several measurements were performed at the line (in the plant of Leonardo Ceramica 1502), by manually taking samples from the belt and disposing them on the measurement plate. However, this procedure can be easily implemented on line, in such a way as to have automatically performed both the sampling and the measurement steps. The developed tool seems thus suitable for the atomising process control.

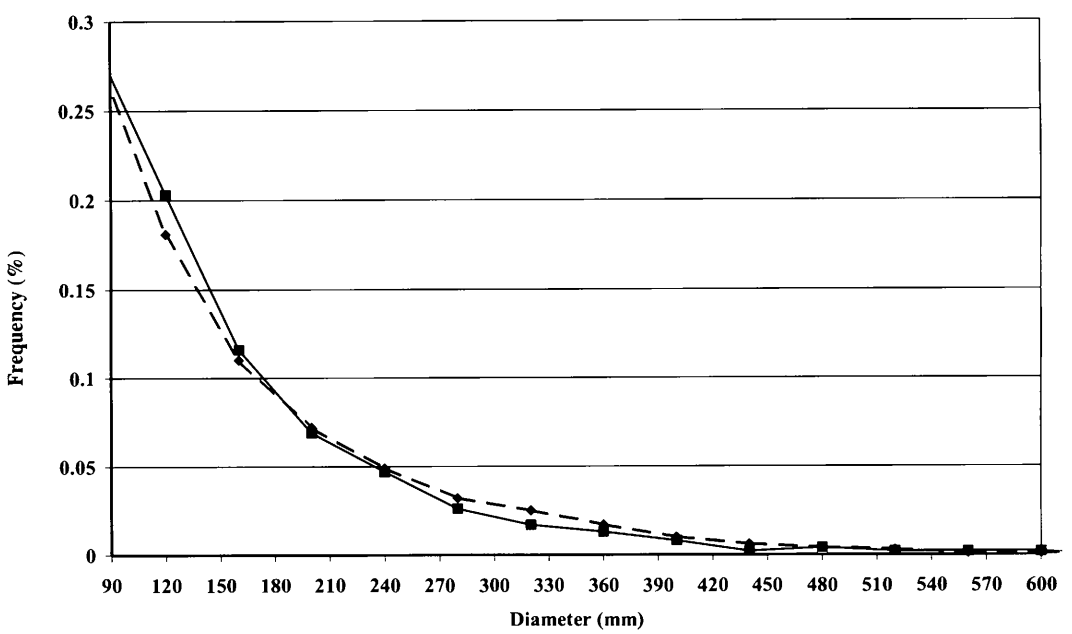

Figure 5. Typical measurement results on powder for porcelain tile production.

5. UNCERTAINTY ANALYSIS

The analysis of measurement system shows three elements that considerably influence measurement results.

5.1 RESOLUTION OF THE OPTICS

Photographic objectives have limited resolution due to diffraction; in practice there is a minimum separation distance between two objects in order to distinguish one from the other [Paone, 1989]. Even in the case of large particles, diffraction manifests itself at the particle edge. Considering a spherical object, the diameter of its image can be computed as:

$$d_i = \sqrt{M^2 \cdot d_p^2 + d_d^2 + d_r^2} \qquad \text{[Eq. 3]}$$

where:

- M is the magnification ratio, 1 in this case;
- d_p is the real particle diameter;
- d_d is the diameter of the diffraction limited image of o point source;
- d_r is the pixel dimension, which has been computed as the diameter of a circle having area = 1 pixel^2 (if we have 9 μm /pixel then d_r =8.5 μm).

With regard to d_d its value can be computed as:

$$d_d = 2.44 \cdot (M+1) \cdot f\# \cdot \lambda \qquad \text{[Eq. 4]}$$

where:

- $f\#$ = 2.8 is the objective numerical aperture used;
- λ is the light wavelength (it has been considered λ =0.6μm even if we have white light).

Therefore, d_d = 8.2 μm .

Having established these values for the parameters, it was possible to estimate the difference between real image diameter d_p and its image diameter d_i. The difference $d_i - d_p$ in the range of particle sizes of interest in this application is always less than 1 μm , that is less than 1 %, which is acceptable.

5.2 OPTICAL ABERRATIONS OF THE OBJECTIVE

Several optical aberrations affect all lens systems. Geometric aberrations can be limited by choosing proper objectives, such as macro-lenses. When operating with white light chromatic aberrations also appear. In order to keep these effects as limited as possible, a macro-objective with a focal length equal to 60 mm has been used in all the tests; this type of objective keeps adequate performance even at its maximum aperture, f#=2.8, used in most of our experiments.

5.3 UNCERTAINTY DUE TO IMAGE PROCESSING

Spatial sampling of the image performed by the digital acquisition system limits spatial resolution. This fact, coupled to the use of a threshold in order to have a binary image needed for particle morphological analysis, causes uncertainty. Such an effect appears on the pixels at the particle edge. Particle diameter is measured, according to

Heywood definition, through an area measurement, which is measured in number of pixels; the uncertainty on area then affects the measured diameter. Particle area has a minimum and a maximum value which derives from the computation of area taking into account border pixels or not; border pixels have an equal probability of being part of the particle or not. Therefore it can be said that the real area will lie between the limits set by the number of border pixels.

Therefore, using uncertainty theory, the measured area can be estimated as [Figliola & Beasley, 1995]:

$$A_m = A_s \pm \frac{P}{2} \qquad \text{[Eq. 5]}$$

where:

A_m = measured area in number of pixels;

A_s = number of pixels of the internal area;

P = number of pixels on the border

In general:

$$D_m = D_s \pm \delta D \qquad \text{[Eq. 6]}$$

Using A_m the Heywood diameter can be computed as:

$$D_m = \sqrt{\frac{4 \cdot A_m}{\pi}} \qquad \text{[Eq. 7]}$$

From Eq. 7 the uncertainty on the Heywood diameter can be expressed as:

$$\delta D = \left.\frac{\partial D}{\partial A}\right|_{A=A_s} \delta A \qquad \text{[Eq. 8]}$$

$$\delta D = \frac{P}{2\sqrt{\pi \cdot A_s}} \cdot \qquad \text{[Eq. 9]}$$

Relative uncertainty can therefore be computed as:

$$i_D = \frac{\delta D}{|D_s|} \qquad \text{[Eq. 10]}$$

and from Eq. 9 and 10:

$$i_D = \frac{P}{4A_s} \qquad \text{[Eq. 11]}$$

Based on the above equations, absolute and relative uncertainty δD and i_D have been computed for each particle diameter. Figure 6 shows the theoretical and experimental values.

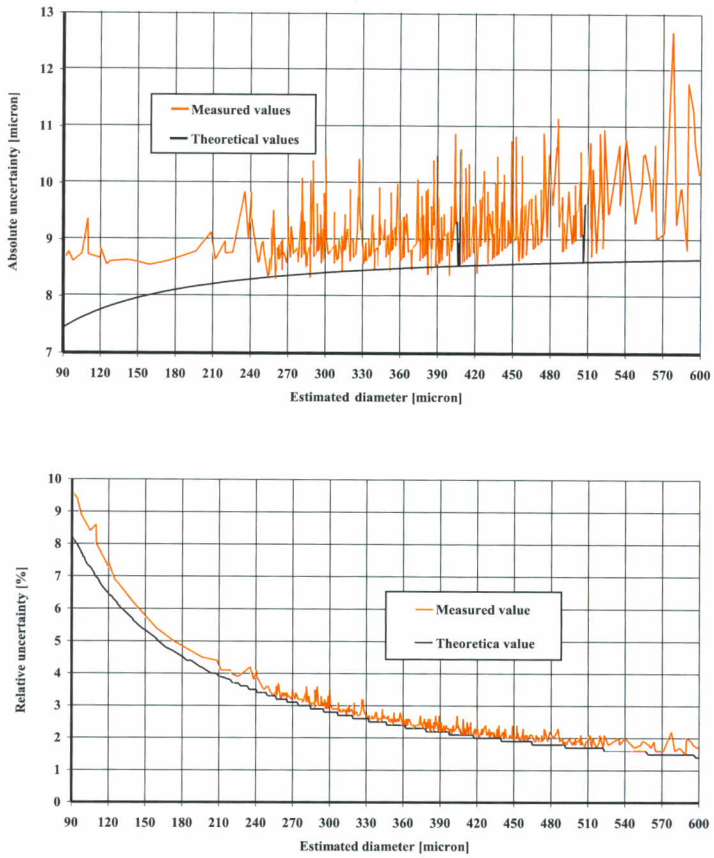

Figure 6. *Absolute and relative uncertainty vs. particle diameter (theory and experiments).*

The following comments can be drawn from Fig. 6:

- Relative uncertainty decreases with D;
- Absolute uncertainty increases slightly with D, with an asymptotic trend up to 200μm ;
- Theoretical curves are the lower limit to uncertainty, because they are computed under the assumption of spherical particles, which have the minimum perimeter for a given area. In reality the shape is not spherical, therefore their image is not a circle and hence experimental estimates of uncertainty provide larger values with scattered results.

As a general result, the uncertainty due to image digitizations and processing is maximum 10% (9μm) for the smallest particles and about 2% (11μm) for the larger ones.

Sub-pixel interpolation can reduce such values. This is done by interpolating image edges with a two-dimensional function, $IG^*(x,y)$, in the image intensity domain. Eq. 12 shows this function:

$$\delta D_T(D) = \sqrt{\delta D_e^2 + \delta D_d^2} \qquad \text{[Eq. 12]}$$

Any pair of pixels along x or y is interpolated with a linear function, which is continuous. The image is then resampled with smaller pixels, so that a higher resolution image is obtained. Figure 7 reports uncertainty after sub-pixel interpolation.

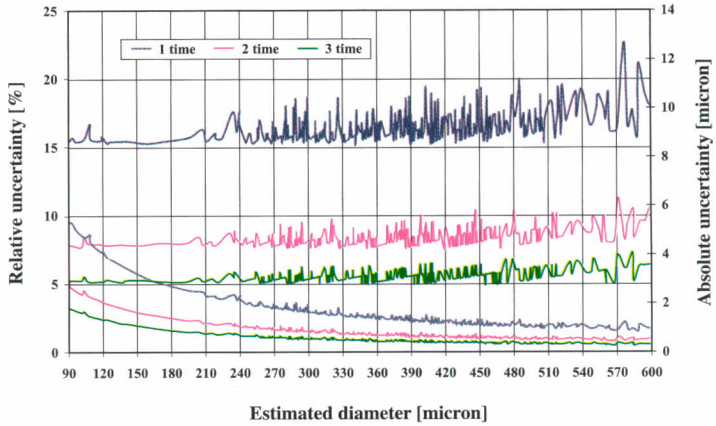

Figure 7. Uncertainty with varying sub-pixel interpolation.

5.4 OVERALL UNCERTAINTY

In order to compute overall uncertainty, all the above contributions need to be taken into account. Equation 13 combines the effects:

$$\delta D_T(D) = \sqrt{\delta D_e^2 + \delta D_d^2} \qquad \text{[Eq. 13]}$$

where, δD_e e δD_d are the contribution of image acquisition and processing and of diffraction respectively.

Relative uncertainty therefore becomes:

$$i_T = \frac{\delta D_T}{D} \qquad \text{[Eq. 14]}$$

Uncertainty due to diffraction is less important than that related to digital image acquisition and processing.

Sub-pixel interpolation provides advantages up to a zoom factor equal to 3x. In this case uncertainty lies between $2\mu m \div 4\mu m$. If the zoom factor is increased, then diffraction effects dominate, keeping minimum uncertainty in the order of 1%. Taking into account that sub-pixel interpolation is relatively time consuming, a zoom equal to 3x is an acceptable compromise.

Figure 8 summarizes the results.

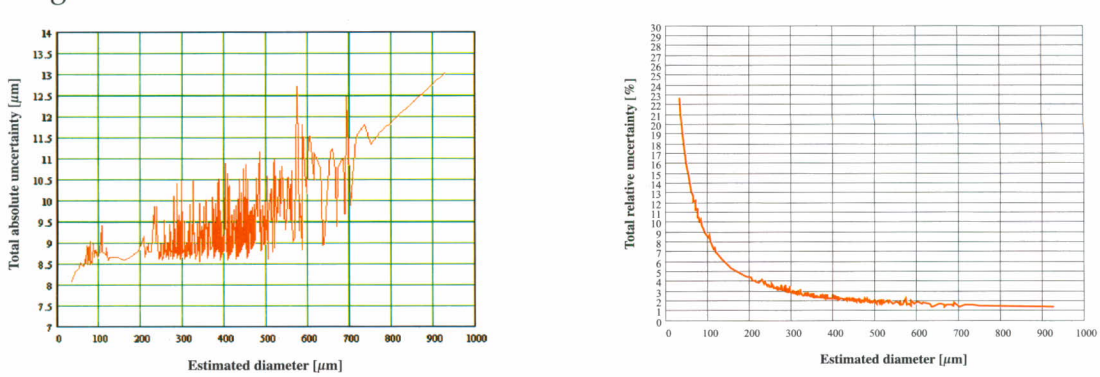

Figure 8. Total absolute and total relative uncertainty of the measurement.

6. CONCLUSIONS

In this paper the development of a ceramic particle size measurement technique based on Computer-Aided Image Analysis (CAIA) has been developed, which should be much quicker than image analysis by microscopy, much more accurate than industrial sieving and less costly than laser diffraction. Another main advantage of this technique is the possibility of on-line particle size control at the exit of the spray dryer, in order to give a quick feedback to the spray-drying process.

The hardware measurement chain has been developed, based on a high-resolution monochrome CCD camera (1kx1k pixels) equipped with a 60-mm macro objective mounted on an extension bellows. The image processing software has been developed and measurement tests have been performed. The acquired image has been conveniently processed before any measurement is done, because the particles must be highlighted with respect to the background. For this reason, appropriate software under the National Instruments LabView environment has been developed, using the Imaq-Vision image-processing library. Once the measurement is performed, the particles are returned to the belt and a new sample, extracted from the line, is analyzed. The measurement, taking a few seconds to complete, must be continuously repeated to generate statistically significant results every few minutes. The dimension range of the particles being measured (90-600 μm) compels us to be close to the scene in order to reducing the measurement uncertainty. At the minimal focal distance the camera can achieve, a pixel is equivalent to 9 μm and this is therefore the error on the particle border. Another problem is that only a few particles at a time could be measured. Therefore, the best compromise between measurement precision and statistical reliability of the results is the key concept to the solution of the problem. The uncertainty of the measurement technique is discussed and laboratory and on-line results are reported.

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