

SIZE DISTRIBUTION OF METALLIC POWDERS : A COMPARISON OF DIGITAL IMAGING AND LASER DIFFRACTION

M.P. GREGOIRE¹⁾, F. MICHEL¹⁾, F. CAMPANA²⁾ & E. PIRARD¹⁾

1) GeMMe-Georessources Minérales et Imagerie Géologique, Université de Liège, Belgium
2) Advanced Coating S.A., Belgium

Introduction

Automated image analysis of particles under controlled orientation (SIA) is becoming a challenging technique for laser diffraction (LD) in the field of sizing metallic particles above 5 μm . Thanks to optimal particle dispersion and fully automated microscopic imaging, it is now possible to gather individual measurements on thousands of particles within a minute. The aim of this paper is to compare results obtained with both image analysis and laser diffraction from a selection of powders (figure 1) in the range between 5 μm and 250 μm .

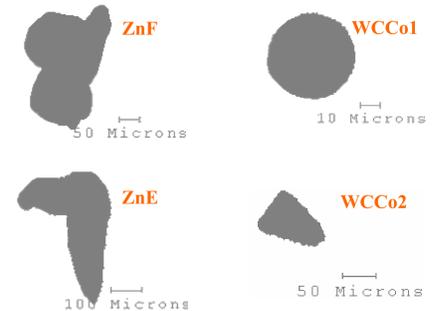


FIGURE 1 : typical median size/median elongation particles of each of the 4 samples.

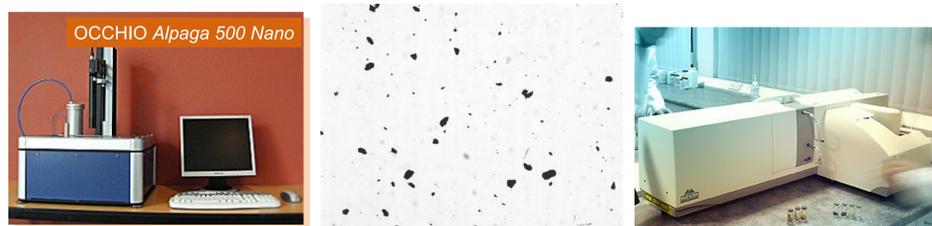
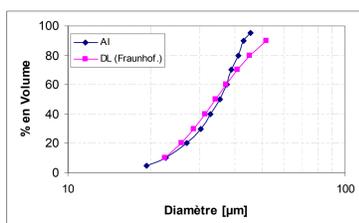


FIGURE 2 : Alpage 500 nano from Occhio (left), image of particles dispersed with Occhio VDD270 on a glass slide (center) and malvern mastersizer 2000 (right).



| % by Volume | SIA | LD | LD (manufacturer) |
|-------------|-------|-------|-------------------|
| 2 | | | 22,19 |
| 5 | 19,19 | 20,21 | |
| 20 | 26,88 | 25,70 | |
| 50 | 35,52 | 34,13 | 33,15 |
| 80 | 41,28 | 45,21 | |
| 95 | 45,6 | 57,57 | 44,71 |
| N | 24250 | N.A. | N.A. |

FIGURE 3 : curves (left) and figure of the volume weighted size distribution for powder WCCO1.

Results & Conclusions

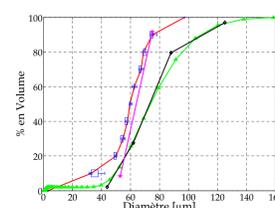
Results for opaque WCCo1 particles (FIGURE 3) show a high correlation. The discrepancy between both techniques appears to be in the range of what is observed among laser diffraction instruments from different manufacturers. For the 3 others sample powders (FIGURE 4), the difference between static image analysis and laser diffraction is bigger. The main reason is the influence of particle shape on the laser diffraction size distribution. Indeed, SIA gives results closer to the sieving data for the well calibrated WCCo2 powder. Both laser diffraction instruments seems to be affected by the median aspect ratio corresponding to 2:1. A comparison of the ZnE and ZnF size distributions shows that laser diffraction is hampered by elongated particles. A second reason explaining the difference between the two techniques is the presence of outliers when the analysis refers to a poorly calibrated material. Just as for any statistical analysis, SIA allows to retrieve the occasional very large particle.

This leads to the following conclusions.

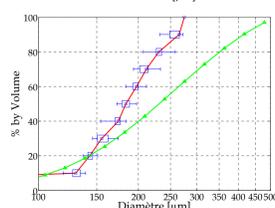
The results shows that SIA is a more robust method towards elongated particles and outliers. It confirms [1] the correlation between SIA and sieving.

Material & Methods

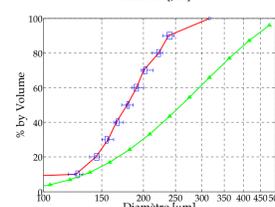
We first selected 4 samples of metallic powders based on their shape and their size range. The typical median size and median elongation particles of each sample are illustrated in the FIGURE 1. Laser diffraction were performed with the malvern mastersizer 2000 coupled with the dispersion unit hydro 2000S (FIGURE 2). Static image analysis results come from Occhio Alpage 500 nano coupled with the dry dispersor Occhio VDD 270 (FIGURE 2). The size is expressed as the diameter of the maximum inscribed disc D_{IN} [1]. The results were analysed statistically with outliers (agregates, sample pollution...) removal and confidence interval computation [2].



| % by Volume | SIA | LD Malvern | LD Microtrac | Tamisaige Labo. | |
|-------------|-------|------------|--------------|-----------------|------|
| 10 [µm] | 32,91 | 49,08 | 2,13 [µm] | 44 | N.A. |
| 20 [µm] | 50,4 | 56,60 | 8,34 [µm] | N.A. | 53 |
| 50 [µm] | 59,55 | 74,00 | 27,48 [µm] | 62 | N.A. |
| 80 [µm] | 68,95 | 96,01 | 79,56 [µm] | 88 | N.A. |
| 90 [µm] | 75,22 | 108,93 | 91,67 [µm] | N.A. | 75 |
| N | 10000 | N.A. | 97 [µm] | 125 | N.A. |



| % by Volume | SIA | LD |
|-------------|--------|--------|
| 10 [µm] | 129,48 | 108,85 |
| 20 [µm] | 142,68 | 142,26 |
| 50 [µm] | 183,3 | 231,17 |
| 80 [µm] | 230,44 | 352,42 |
| 90 [µm] | 265,55 | 415,15 |
| N | 1500 | N.A. |



| % by Volume | SIA | LD |
|-------------|--------|--------|
| 10 [µm] | 125,2 | 133,22 |
| 20 [µm] | 143,98 | 168,36 |
| 50 [µm] | 178,64 | 260,92 |
| 80 [µm] | 222,23 | 378,94 |
| 90 [µm] | 237,88 | 436,14 |
| N | 1500 | N.A. |

FIGURE 4: volume weighted size distribution for WCCo2 (upper), ZnF (middle) and ZnE (lower) obtained with SIA (□), laser diffraction Malvern (▲), laser diffraction Microtrack (◆) and sieving in our laboratory (*).

References

- [1] PIRARD E., VERGARA N. & CHAPEAU V., Direct estimation of sieve size distributions from 2-D image analysis of sand particles, *Proceeding of International Congress for Particle Technology Nuremberg (Germany)*, 2004.
- [2] GREGOIRE M. P., DISLAIRE G. & PIRARD E., Accuracy of size distributions obtained from single particle static digital image analysis, *Proceedings of the International Congress for Particle Technology Nuremberg (Germany)*, 2007.