Data Article

Homogeneity of ball milled ceramic powders: Effect of jar shape and milling conditions

M. Broseghinia a, M. D’Incau a,*, L. Gelisio a, N.M. Pugno b,c,d, P. Scardi a

a Department of Civil, Environmental & Mechanical Engineering, University of Trento, via Mesiano, 77, 38123 Trento, Italy
b Department of Civil, Environmental & Mechanical Engineering, Laboratory of Bio Inspired and Graphene Nanomechanics, University of Trento, via Mesiano, 77, 38123 Trento, Italy
c Center for Materials and Microsystems, Fondazione Bruno Kessler, Via Sommarive 18, 38123 Povo (Trento), Italy
d School of Engineering and Materials Science, Queen Mary University of London, Mile End Road, London E1 4NS, United Kingdom

A R T I C L E I N F O

Article history:
Received 29 June 2016
Received in revised form
20 October 2016
Accepted 17 November 2016
Available online 24 November 2016

A B S T R A C T

This paper contains data and supporting information of and complementary to the research article entitled “Effect of jar shape on high-energy planetary ball milling efficiency: simulations and experiments” (Broseghini et al.,) [1]. Calcium fluoride (CaF2) was ground using two jars of different shape (cylindrical and half-moon) installed on a planetary ball-mill, exploring different operating conditions (jar-to-plate angular velocity ratio and milling time). Scanning Electron Microscopy (SEM) images and X-Ray Powder Diffraction data (XRPD) were collected to assess the effect of milling conditions on the end-product crystallite size. Due to the inhomogeneity of the end product, the Whole Powder Pattern Model (WPPM, (Scardi, 2008) [2]) analysis of XRPD data required the hypothesis of a bimodal distribution of sizes – respectively ground (fine fraction) and less-to-not ground (coarse fraction) – confirmed by SEM images and suggested by the previous literature (Abdellatif et al., 2013) [3,4]. Predominance of fine fraction clearly indicates optimal milling conditions.

© 2016 The Authors. Published by Elsevier Inc. This is an open access article under the CC BY license (http://creativecommons.org/licenses/by/4.0/).

DOI of original article: http://dx.doi.org/10.1016/j.matdes.2016.06.118
* Corresponding author.
E-mail address: mirco.dincau@unitn.it (M. D’Incau).

http://dx.doi.org/10.1016/j.dib.2016.11.070
2352-3409 © 2016 The Authors. Published by Elsevier Inc. This is an open access article under the CC BY license (http://creativecommons.org/licenses/by/4.0/).
## Specifications Table

| Subject area               | Physics, Chemistry                     |
|----------------------------|----------------------------------------|
| More specific subject area | Materials Science, Powder technology   |
| Type of data               | Images, graphs, raw and analyzed data  |
| How data was acquired      | SEM: ESEM FEI XL30 XRPD: Rigaku PMG/VH diffractometer |
| Data format                | Raw, analyzed                          |
| Experimental factors       | Data collected on milled sample without any pre-treatment. Non-destructive tests. |
| Experimental features      | SEM micrographs: standard Secondary Electron detector |
|                            | XRPD data: CuKα radiation, scintillation counter detector and bent graphite analyzer crystal. Slits: DS 1°, Soller 2° slits, SS 1°, secondary Soller 2°, RS (0.15 mm). Instrumental profile measured using NIST SRM 660a (LaB₆ powder) |
| Data source location       | Trento (via Mesiano, 77), Italy, 46°04'N 11°07'E |
| Data accessibility         | All data are with this article          |

## Value of the data

- The planetary ball-milling process is ubiquitous in the production of nanostructured materials and modification of their properties. The choice of optimal operating conditions defines end product characteristics. Data reported in this manuscript guide the understanding of the effect of two milling parameters (jar-to-plate angular velocity ratio and milling time) on the dimensional characteristics of the end product.
- The assessment of the milling behavior (e.g. coexistence of fine and coarse fractions and their distributions) of a new jar design and the comparison with the standard cylindrical one can be drawn from reported data and clearly show the importance of the vial shape on the end product properties and on comminution efficiency.
- XRPD raw data could be modeled with different approaches and/or used to extract more information on e.g. powder homogeneity and defects content introduced by the severe deformation (see [1,6]).

## 1. Data

Fig. 1 illustrates a representative case of optimal modeling of XRPD data (Supplementary 1 and 2) by a WPPM method, which requires two lognormal distributions of crystallite domain sizes describing respectively a finely ground and a coarse fraction. The validity of this hypothesis, already suggested by [3,4], is further demonstrated by SEM pictures (selected cases reported in Fig. 2), clearly showing the coexistence of grains characterized by considerably different sizes. Fig. 3 compares end products size distribution obtained by WPPM analysis of data from samples milled with the cylindrical and half-moon jars for different milling times.

Supplementary 1 reports raw and analyzed XRPD data for different jar-to-plate velocities (reported for a representative case in Fig. 1; results of data analysis reported in Fig. 8 in [1]).

Supplementary 2 reports raw and analyzed XRPD data for different milling times (results of data analysis reported in Fig. 3).
2. Experimental design, materials and methods

2.1. Materials

Calcium fluoride (CaF$_2$) from CARLO ERBA Reagents S.r.l.

2.2. Milling

Samples were milled in a Fritsch Pulverisette 4 (P4) planetary ball-mill under different operative conditions (jar-to-plate angular velocity ratio and milling time). Twelve balls were inserted in a cylindrical and in a half-moon jar (physical and geometrical properties reported in [1,6]), designed by the authors and produced at the University of Trento (Italy).
2.3. Data acquisition

An ESEM FEI XL 30 was employed to acquire SEM images while XRPD data were collected using a Rigaku PMG/VH diffractometer according to the procedure reported in [1,5].

2.4. XRPD data analysis

WPPM analyses [2] were performed using the software PM2K [7] and details are reported in [1].
Acknowledgements

The present work was partly supported by Fondazione Caritro, under Project No. 2013-0247 Mechanical activation to improve bioavailability and reduce adverse effects of drugs in the therapy of chronic diseases. NMP is supported by the European Research Council (ERC StG Ideas 2011 BIHSNAM n. 279985, ERC PoC 2015 SILKENE nr. 693670), by the European Commission under the Graphene Flagship (WP14 Polymer Composites, No. 696656).

Transparency document. Supplementary material

Transparency data associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.dib.2016.11.070.

Appendix A. Supplementary material

Supplementary data associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.dib.2016.11.070.
References

[1] M. Broseghini, M. D’Incau, L. Gelisio, N. Pugno, P. Scardi, Effect of jar shape on high-energy planetary ball milling efficiency: simulations and experiments, Mater. Des. 110 (2016) 365–374. http://dx.doi.org/10.1016/j.matdes.2016.06.118.
[2] P. Scardi, Microstructural properties: lattice defects and domain size effects, in: R. Dinnebier, S. Billinge (Eds.), Powder Diffraction. Theory and Practice, RSC Publishing, Cambridge, 2008, pp. 378–416.
[3] M. Abdellatif, M. Abele, M. Leoni, P. Scardi, Solid state nuclear magnetic resonance and X-ray diffraction line profile analysis of heavily deformed fluorite, Thin Solid Films 530 (2013) 44–48. http://dx.doi.org/10.1016/j.tsf.2012.09.020.
[4] M. Abdellatif, M. Abele, M. Leoni, P. Scardi, Combined X-ray diffraction and solid-state 19F magic angle spinning NMR analysis of lattice defects in nanocrystalline CaF2, J. Appl. Crystallogr. 46 (2013) 1049–1057. http://dx.doi.org/10.1107/S0021889813016944.
[5] M. D’Incau, High Energy Milling in Nanomaterials Technologies: Process Modelling and Optimization (Doctoral thesis), University of Trento, Trento, 2008.
[6] M. Broseghini, L. Gelisio, M. D’Incau, C.L. Azanza Ricardo, N. Pugno, P. Scardi, Modeling of the planetary ball-milling process: the case study of ceramic powders, J. Eur. Ceram. Soc. 36 (2016) 2205–2212. http://dx.doi.org/10.1016/j.jeurceramsoc.2015.09.032.
[7] M. Leoni, T. Confente, P. Scardi, PM2K: a flexible program implementing whole powder pattern modelling, Z. für Krist. Suppl. 23 (23) (2006) 249–254. http://dx.doi.org/10.1524/zksu.2006.suppl_23.249.