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9th International Conference on Mechanochemistry and Mechanical Alloying (INCOME 2017)

Novel techniques to prepare materials for pharmaceutical, medical and other applications

Reviewed by Jose Villoria
Johnson Matthey, Blounts Court, Sonning Common, Reading, RG4 9NH, UK

Email: jose.villoria@matthey.com

Introduction

The 9th International Conference on Mechanochemistry and Mechanical Alloying was held from 3rd–7th September 2017, in Košice, Slovakia. There were approximately 180 delegates from a wide range of affiliations including European academic institutions (Italy, Spain, France, Germany, Poland, Hungary, Slovakia, Slovenia, Croatia, UK, Russia and others); but also from the USA, Mexico, India, Australia, Japan and Singapore. This conference was the ninth in a series of international conferences on mechanochemistry held every three years since its beginning in 1993 in Košice, Slovakia.

The present report aims to show the main topics of the conference and summarises a few of the contributions.

Mechanochemical Synthesis Techniques

'A Quest for Mechanisms of Mechanically Activated Transformations' by F. Deloglu (Università degli Studi di Cagliari, Italy) showed mechanochemistry work with a shaker mill. It included an analysis of the acoustics of the mill during operation and the possibility of identifying the impact and rolling stages of the ball inside the vessel. Deloglu also explained that the refinement of metals under milling action is an exponential function of time. The exponent measures the rate of refinement and depends on the temperature, volume of the vessel and characteristics of the metals. Another result showed the compact ratio (cold-welding) of bismuth, tungsten, tellurium and other metals during milling as an inverse function of their melting point. Highlights were the use of acoustic measurements to understand the milling process; the compaction of metals related to their melting point and an invitation to follow the European Cooperation in Science and Technology (COST) programme.

'Chalcogenide Quaternary Nanocrystals for Solar Cells: Mechanochemical Synthesis and Properties of Kesterite, Cu_2ZnSnS_4' by P. Baláž (Slovak Academy of Sciences, Bratislava, Slovakia) showed the production of kesterite-type structures with formula Cu_2ZnSnS_4 by milling pure copper, zinc, tin and sulfur powders with tungsten carbide media under a preventive (inert) atmosphere. The results showed the initial formation of CuS (covellite) under milling followed by CuZnS and finally the target Cu_2ZnSnS_4 kesterite. Attempts to scale this reaction up used an industrial eccentric vibration mill with tungsten carbide media at the University of Clausthal. Baláž explained that the temperature increase during milling is quite moderate and several kilos per batch can be produced in such a mill.

'Chemistry 2.0: Developing a New, Solvent-Free System of Chemical Synthesis Based on Mechanochemistry' by T. Friščić (Department of Chemistry at McGill University, Montreal, Canada) showed the monitoring of mechanochemical reactions for the synthesis of metal-organic frameworks (MOFs). Another reaction described was solvent-free assembly of Bi_2S_3 nanoparticles using different ammine ligands. Other work by Friščić included the low energy saccharification of biomass using different organic molecules. The highest yield was achieved with cellulose with a 35% maximum yield (1–3).

'Solvent-Free Mechanochemical Synthesis – Fundamentals, Scale-up and Commercialisation' by S. L. James (School of Chemistry and Chemical Engineering, Queen's University of Belfast, UK) showed the formation of a zinc imidazolate framework by milling ZnO and imidazole in the presence of dimethylformamide: the reaction rate is dependent on the milling energy and does not increase with increasing temperature. He also showed the different steps of the reaction and demonstrated first-order kinetics. Finally, James introduced the spin-off company MOF Technologies™ which produces MOFs at a rate of 15 kg h^{-1}. He also introduced MOF materials with application to improve the shelf life of fruit.

'Synthesis by Twin Screw Extrusion (TSE)' by D. Crawford (School of Chemistry and Chemical Engineering, Queen's University of Belfast, UK) showed a twin screw extruder used to make a commercial MOF at industrial scale. It consists of two feeders at different positions along a couple of attached screws which are rotating (**Figure 1**) (4).

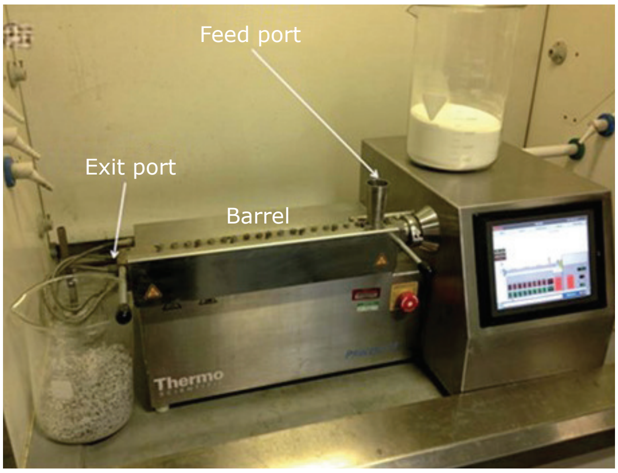

Fig. 1. Twin screw extruder with key parts highlighted. The two screws which convey and knead the reactants are housed in the barrel (4). Published by The Royal Society of Chemistry

The screws have sections to convey the powder or to shear it. The final product is obtained at the end of the screws in powder form. Another example was the processing of amorphous paracetamol with citric acid (50% each) at 100°C.

'Mechanochemical Synthesis of Highly-Ordered Microporous Zirconium-Based Metal-Organic Frameworks' by K. Užarević (Department of Physical Chemistry, Laboratory for Green Synthesis, Ruđer Bošković Institute, Zagreb, Croatia) presented the synthesis of different MOFs by mechanochemistry as part of a new International Fellowship Mobility Programme for Experienced Researchers in Croatia (NEWFELPRO) project with a Marie Curie grant from the European Union 7th Framework Programme. One of these MOFs was UiO-66 which was made by milling the reactants in the presence of methanol vapours at 45°C. Another MOF formulation prepared by milling was NU-1000. In summary, Užarević showed that mechanochemistry is suitable to make large pore MOFs.

'Spark Plasma Sintering of Mechanically Milled Powders: Gaining Advantages from a Combination of Two Non-Equilibrium Powder Processing Techniques' by D. V. Dudina (Lavrentyev Institute of Hydrodynamics SB RAS, Novosibirsk, Russia) presented spark plasma sintering (SPS) equipment using discontinuous current pulses with uniaxial pressure. The voltage used was 5 V. The technique uses a high heating rate and short sintering time with relatively low pressures (50–100 MPa). It operates under reducing atmospheres so can reduce oxides and form carbides by reaction with graphite. The general advantage of SPS to produce carbides is an increase in hardness of mechanically alloyed composites by incorporation of carbon into the structure. The material shown as an example was a titanium carbide composite with formula Ti_3SiC_2-Cu made by rapid sintering of fine grain materials. The SPS process can be also applied to create porous materials.

'Microstructure, Porosity and Wear Resistance of New Ti–10Ta–8Mo (wt%) Biomedical Alloy Prepared by High-Energy Ball Milling and Annealed Processes' by I. Matuła (Institute of Materials Science, University of Silesia in Katowice, Katowice, Poland) presented the use of planetary ball milling for the formation of titanium alloys with application in medical devices. After the milling a cold-pressing process was applied followed by annealing at 950°C for 24 h and 450°C for 3 h. During annealing an increase of the β-phase of Ti is observed and this depends on the particle size distribution. Further transformation to this β-phase is supposed to be hindered by the presence of the α-phase at the core of the Ti crystals. The decrease in the porosity of the initial material with milling time was also shown. Nevertheless, the presence of interconnected pores was observed in samples milled for 15 h.

'The Effect of Sn as the Process Control Agent on the Fabrication and Structural Properties of New Ti–Ta–Mo–Sn Biomedical Alloy Synthesised by High Energy Ball Milling' by G. Dercz (Institute of Materials Science, University of Silesia in Katowice, Katowice, Poland) presented new Nitinol formulations for medical components, including Ti-Mo-Ta and the addition of Sn. The formation of the Ti-Mo-Ta alloy was carried out by planetary milling. Doping with 1–5 wt% Sn proved crucial to decrease the cold-welding effect during milling of the metal powders. The optimum doping was 3 wt% Sn.

Other interesting work included the field assisted sintering technique (FAST) presented by D. V. Dudina; pulse plasma sintering (PPS) presented by A. Miklaszewski (Poznań University of Technology, Poland); electrical discharge assisted mechanical milling (EDAMM) by A. Calka (University of Wollongong, Australia) and electrical resistance sintering (ERS) by E. Caballero (Universidad de Sevilla, Spain). *In situ* monitoring of mechanochemical processes was also presented. Some of the contributions characterised the evolution of the powder during milling by extended X-ray absorption fine structure (EXAFS) and synchrotron techniques. Continuous analysis of gas compositions from a vessel during milling under a reactive atmosphere and evaluation of the catalytic activity of the milled powder *in situ*.

Conclusions

During the INCOME 2017 conference, scientists from many countries showed their latest work on the use of mechanochemistry to synthesise a wide range of materials including: sulfides; glassy materials such as Li-La-Zr oxides for solid electrolytes; pharmaceutical co-crystals; MOFs; metal alloys and composites. Some presentations included studies of the synthesis mechanisms, amorphisation of powders or recrystallisation. Other contributions presented new advances by using 'sintering' techniques in combination with milling processes. Some of these methods can produce nitrides, carbides and other challenging materials with enhanced properties.

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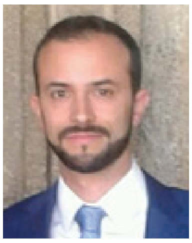

Jose A. Villoria is a Senior Scientist at Johnson Matthey, Sonning Common, UK. He received his BEng degree in Chemical Engineering from the University of Salamanca, Spain, and PhD in Physical Chemistry from the Autonomous University of Madrid, Spain, with a thesis on heterogeneous catalysis. Since then his research activity has focused on the production of different materials by mechanochemistry and understanding the mechanisms of mechanochemical processes.

