Applications of electron microscopy in mechanochemistry

Bahman Nasiri-Tabrizi1, Saeid Baradaran1, Erfan Zalnezhad2 and Wan Jefrey Basirun3

1 Department of Mechanical Engineering, Faculty of Engineering, University of Malaya, 50603 Kuala Lumpur, Malaysia
2 Center of Advanced Manufacturing and Material Processing, Department of Mechanical Engineering, Faculty of
Engineering, University of Malaya, Kuala Lumpur 50603, Malaysia
3 Department of Chemistry, Faculty of Science, University of Malaya, 50603 Kuala Lumpur, Malaysia

Mechanochemistry is one of the most important fields of solid-state chemistry which is intensively developed during the last decade. Depending on the processing conditions, the mechanochemical processes fall into two categories: progressive reaction (PR) and mechanically induced self-sustaining reaction (MSR). In the first mode, the reaction may extend to a very small volume during each collision, resulting in a gradual transformation. In the second manner, if the reaction enthalpy is sufficiently high, a self-propagating combustion reaction can be initiated after a certain time. The simplicity, reproducibility, and low processing cost are the main advantages of this method. Therefore, the mechanochemical process can be served, when the mass production of nanopowders is required. From the microstructural point of view, the mechano-synthesized powders usually possess a well-defined structure due to the perturbation of surface-bonded species. However, the determination of the morphological features is generally one of the most difficult problems in powder technology because there is no general shape factor available that clearly differentiates all possible kinds of shapes. In the present review, the applications of electron microscopy in the morphological characterization of the mechano-synthesized nanopowders were investigated. Results showed that how the morphological features of the products can be effectively controlled by varying the processing parameters.

Keywords Morphological features; Mechanochemical; Electron microscopy (SEM/FE-SEM/FE-STEM/TEM); MSR; Nanopowders

1. Mechanosynthesis of nanostructured materials

One of the most important aspects of nanotechnology is the modification and design of solids to obtain functionalized materials with tailored properties [1]. Hence, numerous attempts have been made to develop new nanomaterials with desirable properties [2–5]. In general, the top down (break or dissociate solids into finer) and the bottom up (assemble atoms together) approaches are used to produce nanostructured materials [6]. These attitudes involve various types of synthesis routes. In each method, processing conditions can be varied across a wide range, resulting in several sub-methods (Fig. 1). With such a wide variety of production methods, choosing an appropriate route considering the intended application is necessary. For example, in the field of bioceramics (nanoapatites), it has been recently reported that around 25% of the total 650 papers indexed over the period of 1999–2011 are solely connected to the conventional chemical precipitation method. Following chemical precipitation, combination processes, the hydrothermal approach, and sol-gel method are the next most well-known methods of preparing nanoapatites [7].

Fig. 1 Different methods along with their sub-groups for the preparation of nanomaterials.
According to the literature [7–17], the synthetic nanomaterials had different shapes and dimensions depending on the production method and its variations. For instance, some typical morphologies of nanohydroxyapatite (n-HAp) produced by the conventional methods are presented in Fig. 2.

As shown in this figure, n-HAp with various morphologies can be generated by different methods, each of which is of interest in certain applications. It has been proven that the spherical particles are better than other irregular shapes due to the well space fillings and the low percentage of voids in the final product [18]. Moreover, granules with a
smooth spherical geometry are helpful in growth and attachment of bone tissue that improve osseointegration [19,20]. Also, it has been found that the nanostructured apatites with ellipse- or rod-like morphology inhibit the proliferation of malignant melanoma cells [21]. In fact, these nanostructures may be helpful to remedy cancer. Besides, the obtained products could be used as strength enhancing additives for the preparation of bionanocomposites [22].

In general, the advantages of the wet process are that the by-product is almost water [23,24] and the probability of contamination during processing is very low. However, the composition of the product is strongly affected by even a slight difference in the reaction circumstances and the production process is often time consuming, which are the disadvantages in the mass production of nanomaterials. Furthermore, the handling of the materials and the operation of the apparatus are typically complicated which cause poor reproducibility and high processing cost. Therefore, for the large scale synthesis of high crystalline nanomaterials, the dry processes including solid-state method and mechanochemical treatment are more suitable than the wet processes due to their high reproducibility and low processing cost in spite of the risk of contamination during milling. In this attitude, precisely controlled conditions are not necessary; as a result the dry processes are proper for the mass production of nanopowders. Of course among the dry methods, the solid-state approach has received the least attention due to its inherent limitations in synthesizing nanosized particles and its lack of clear control over the microstructural characteristics of the nanopowder [7].

Fig. 3 displays the schematic views of conventional solid-state approaches to prepare nanopowders and dense microstructures. In the first approach in Fig. 3a, the ingredients are initially mixed and then heat treated at high temperatures. In this trend, the phase and chemical purity of the products can vary depending on the type of precursors. It should be mentioned that milling is usually carried out in order to mix the raw materials. During thermal treatment at high temperature, activation of the mixed powder occurs which leads to the formation of a well-crystallized structure. One of the main disadvantages of this method is that the synthesized powder has heterogeneity in its phase composition due to the small diffusion coefficients of ions within the solid phase [25].

In another perspective (Fig. 3b), the commercial grade or previously synthesized powders are first mixed, if necessary, and then are pressed at high pressure. Eventually, the compressed sample is annealed at high temperatures to produce dense microstructures. Moreover, the sintering process can be carried out by hot isostatic pressing (HIP). This process is applied when great uniformity is required, and where net-shape production leads to economic advantages [26].

![Fig. 3](image_url)

On the other hand, mechanochemical process (MP) as one the most important fields of solid state chemistry is intensively developed, so that a large number of reviews and papers published on this subject in the last decades [27–29]. In this method, pressure can be provided via conventional milling equipments, ranging from low-energy ball mills to high-energy stirred mills. In a mill, the raw materials are crushed between the balls and wall (horizontal or planetary ball mill, attritor, vibratory ball mill), or between rings or ring and wall (multi–ring media mill) [30]. Mechanical activation results in the creation of defects in solids, accelerates the migration of defects in the bulk, increases the number of contacts between particles, and renews the contacts. In these circumstances, chemical interaction occurs
between solids [31]. The distinguished features of this technique are that melting is not essential and that the products have nanostructural characteristics. From the chemical reaction point of view, the mechanochemical processes are divided into two categories: progressive reaction (PR) and mechanically induced self-sustaining reaction (MSR). In the first mode, the reaction may extend to a very small volume during each collision, resulting in a gradual transformation. In the second manner, if the reaction enthalpy is sufficiently high, a self-propagating combustion reaction can be initiated after a certain time [1]. A schematic view of mechanochemical processes to prepare nanostructured materials is shown in Fig. 4.

![Fig. 4](image)

**Fig. 4** A schematic view of mechanochemical processes including PR and MSR modes to prepare nanostructured materials (single phase nanopowder, composite structures and intermetallic compounds).

In the next section, a part of the recent developments of nanostructured materials from the mechanochemical processes have been reported. It is well known that the structural and morphological features of nanostructured materials play important roles in specialized applications [32–34]. Therefore, the influence of processing parameters on the formation, structural and morphological characteristics of various nanopowders were discussed. Results of some studies suggest that the size and number of balls had no major effect on the production time and grain size of nanopowders, while decreasing the rotation speed or ball to powder weight ratio (BPR) increased synthesizing time and the grain size of the product [35]. From another point of view, the crystallinity degree, lattice strain, crystallite size, morphology, mean particle size, and the agglomerate size distribution of nanopowders were influenced strongly by the milling time, milling media, milling atmosphere, mixing ratio, chemical composition of reagents, and subsequent thermal treatment [29, 36–39].
2. Morphological features of the mechanosynthesized nanopowders

As a matter of fact, the product of mechanochemical processes is in powder form. Therefore, the mechanosynthesized powders must be analyzed for their size, shape, surface area, phase constitution, and microstructural features. In addition, one could also characterize the transformation behavior of the milled powders in thermal treatment. During the mechanical activation, powder particles are repeatedly flattened, cold welded, fractured and re-welded [27]. In fact, the mechanochemical process contains high internal stress that affects the nanostructural characteristics [40]. Accordingly, the calculation of crystallite size and lattice strain in mechanically alloyed powders by X-ray diffraction (XRD) is very important because the phase constitution and transformation appear to be critically dependent upon them [27,28]. It should be mentioned that not all investigators characterize their products for all of the above mentioned features.

From the microstructural point of view, particle shape analysis is one of the most difficult problems in powder technology. This complexity is due to no general shape factor available that clearly differentiates all possible kinds of shapes [41]. However, the morphological features (size and shape) of the mechanosynthesized powders may be determined accurately using direct methods of either scanning electron microscopy (SEM) for relatively coarse powders or transmission electron microscopy (TEM) for finer powders. Here, an updated review of the morphological features of the mechanosynthesized nanopowders is provided [42–48]. To clarify the size and morphology of the products, the present study focused on SEM/FE-SEM and TEM/FE-STEM analysis.

2.1 Morphological features of nanopowders produced by progressive reaction (PR)

2.1.1 Production of poorly crystalline and crystallized tricalcium phosphate nanopowders

Our recent findings showed that poorly crystalline and crystallized tricalcium phosphate nanopowders can be produced by progressive mode of mechanochemical reaction [42,43]. To this end, calcium hydroxide (Ca(OH)\textsubscript{2}), anhydrous dicalcium phosphate (CaHPO\textsubscript{4}), calcium oxide (CaO) and phosphorous pentoxide (P\textsubscript{2}O\textsubscript{5}) were used as raw materials. The weight ratio of ball-to-powder, total powder mass and rotational speed were 20:1, 6 g, and 600 rpm, respectively. The following reactions were mechanically activated for 10 h using sealed tempered chrome steel vials and balls. After that, to assess the influence of annealing on the phase transformation, the milled powder was filled in a quartz boat, and then heat treated under atmospheric pressure at 1100 °C for 1 h.

\[
\begin{align*}
3\text{CaO} + \text{P}_2\text{O}_5 & \rightarrow \text{Ca}_3(\text{PO}_4)_2.x\text{H}_2\text{O} \quad \text{(Activated under air atmosphere, Ca/P = 1.5)} \\
\text{CaO} + 2\text{CaHPO}_4 & \rightarrow \text{Ca}_3(\text{PO}_4)_2.x\text{H}_2\text{O} \quad \text{(Activated under air atmosphere, Ca/P = 1.5)} \\
3\text{Ca(OH)}_2 + \text{P}_2\text{O}_5 & \rightarrow \text{Ca}_3(\text{PO}_4)_2.x\text{H}_2\text{O} \quad \text{(Activated under air atmosphere, Ca/P = 1.5)} 
\end{align*}
\]

Fig. 5 A schematic view of the formation and morphological features of nano-sized tricalcium phosphate powders [42].
Results indicated that the structural features as well as morphological characteristics of the products were influenced by the chemical composition of reactants. From the XRD data, the crystallite size of the products ranged from about 20 to 28 nm. A schematic view of the formation and morphological features of nano-sized tricalcium phosphate powders are shown in Fig. 5. According to this figure, during the mechanical activation of reaction (2), \( \text{Ca}_2\text{P}_2\text{O}_7 \) and \( \text{Ca(OH)}_2 \) were formed as intermediate phases which eventually resulted in the formation of nano-sized tricalcium phosphate powder. In the reaction (3), the formation of nanopowder was strongly influenced by the presence of \( \text{P}_2\text{O}_5 \) as a source of \( \text{PO}_4^{3-} \) group. This can be explained considering the hygroscopic nature of \( \text{P}_2\text{O}_5 \) which led to the formation of \( \text{H}_3\text{PO}_4 \) at the beginning of milling. Accordingly, the reaction of \( \text{Ca(OH)}_2 \) with \( \text{H}_3\text{PO}_4 \) caused the formation of \( \text{Ca}_2\text{P}_2\text{O}_7 \) as an intermediate phase. Mechanosynthesis progressed by the reaction of \( \text{Ca}_2\text{P}_2\text{O}_7 \) with the remaining \( \text{Ca(OH)}_2 \) which finally resulted in the generation of nano-sized tricalcium phosphate powder. In this reaction, large agglomerates/particles were developed due to the high moisture absorption during the milling process. According to the microscopic observations, the synthesized powders showed cluster-like structures composed of spheroidal particles with an average size of around 23 nm and ellipsoidal particles with a mean size of about 30 nm. During heating at 1100 °C, crystallization of amorphous phase occurred and consequently high crystalline \( \beta \)-tricalcium phosphate \((\beta-\text{TCP})\) was generated. Based on the SEM and TEM observations (Fig. 6), the product showed a bimodal grain size distribution characterized by the presence of coarse grains with a mean size of around 2 \( \mu \)m along with finer grains with an average size of about 300 nm. In a nutshell, the proposed reactions can be used for mass production of nanostructured tricalcium phosphates.

**Fig. 6**  (a) SEM micrograph and (b) TEM image of the product out of reaction (1) after annealing at 1100 °C for 1 h [43].

2.1.2 Preparation of chlorapatite nanopowder

In accordance with our recent findings, chlorapatite nanopowders (n-CAp) can be synthesized by a novel facile mechanochemical process (PR mode) [44,45]. For this purpose, the reagents were \( \text{CaO} \), \( \text{P}_2\text{O}_5 \) and calcium chloride \((\text{CaCl}_2)\). The mole ratio of calcium to phosphorous was equal to 1.67. Milling was carried out in a high energy planetary ball mill at rotational speed of 600 rpm and a ball-to-powder weight ratio of 20:1 using polyamide-6 vials (vol. 125 ml) and Zirconia balls (20 mm in diameter) for 5, 30, 60, 180, and 300 min according to the following reaction.

\[
9\text{CaO} + \text{CaCl}_2 + 3\text{P}_2\text{O}_5 \rightarrow \text{Ca}_{10}(\text{PO}_4)_6\text{Cl}_2
\]

**Fig. 7** A schematic view of the formation and SEM images of n-CAp before and after annealing in the range 900-1300 °C [44].
After the milling process, the mechano-synthesized powder was filled in a quartz boat and then heat-treated in the range 900–1300 °C for 1 h. Results showed that the formation of n-CAp proceeded in several steps. At the beginning of milling, the main products were stoichiometrically deficient chlorapatite and calcium oxide. After that calcium-deficient apatite reacted with the remaining CaO which caused the formation of chlorhydroxyapatite (CHAp, \( \text{Ca}_{10} \left( \text{PO}_4 \right)_{6} (\text{OH})_2 \cdot 2x \text{Cl}_{2x} \)). Finally, high crystalline n-CAp was formed after prolonged milling. By increasing the milling time to 300 min, the lattice strain increased significantly to around 0.0108±0.0005, while the crystalline size declined sharply to about 28±2 nm. During annealing in the range 900–1300 °C, the decomposition of CAp to tricalcium phosphate, tetracalcium phosphate, and hydrochloric acid was dominant. SEM images demonstrated that the milled sample had cluster-like structure which consisted of fine particles with a mean size of about 95 nm. During heating, the coalescence of nanoparticles occurred and eventually a coarse structure with an average grain size of around 3 μm was formed after annealing at 1300 °C.

2.2 Morphological features of nanopowders produced by MSR mode

In self-propagating synthesis, intimate contact between the reactant phases is an essential requirement. In a well-mixed sample, the occurrence of the combustion reaction results in a rapid rise in temperature of the milling medium, and provides the conditions for quick transformation. This type of reaction mechanism can be predicted by calculating adiabatic temperature (\( T_{\text{ad}} \)). It has been reported that the reaction can be a self-sustained combustion, when the adiabatic temperature of the reaction is higher than 1800 K [49].

2.2.1 Production of titanium diboride (TiB₂) nanopowders

Our recent studies showed that titanium diboride nanopowders (n-TiB₂) can be synthesized by MSR mode of mechanochemical reaction [46]. In fact, the influence of milling parameters on the formation of n-TiB₂ was investigated. For this purpose, boron oxide (B₂O₃, mean particles size 30±5 μm), titanium dioxide (TiO₂, < 2 μm) and elemental magnesium (Mg, < 100 μm) were used as the reactants. Besides, sodium chloride (NaCl, analytical grade) was added to the reaction mixture as an inert phase in the synthesis reaction. The milling process was carried out in a high-energy planetary ball mill using hardened chromium steel vials (vol. 125 ml) and balls (15 and 20 mm in diameter) under a high-purity argon atmosphere (99.99 % purity). The rotational speed was 600 rpm. To assess the influence of milling parameters on the formation of n-TiB₂, different ball to powder weight ratios (10:1, 15:1 and 20:1) were chosen. In addition, the total powder mass ranged from 3 to 7 g. To evaluate NaCl catalytic effect on the reduction reaction, the mechanical activation was also executed in the presence of 5 and 10 wt% NaCl. After the milling process, the mechano-synthesized powder with a BPR of 15:1 was leached in 18% HCl aqueous solution at 60 °C for 30 min to remove unwanted phases and other impurities. Results showed that the formation and structural features of titanium diboride nanopowders were notably influenced by ball to powder weight ratio, total powder mass and weight fraction of diluent (NaCl). With BPRs of 10:1, 15:1 and 20:1, a combustion reaction occurred after 73, 34 and 40 min, respectively, which resulted in the formation of TiB₂ as a major compound and MgO and Mg₂TiO₄ as unwanted phases. With a BPR of 15:1, the addition of 5 wt% NaCl delayed the combustion reaction to 60 min and led to the formation of TiB₂, MgO and Mg₂TiO₄ with the crystallite size of around 24, 40 and 45 nm, respectively. After the leaching process, the unwanted phases were completely removed and thus a single-phase n-TiB₂ with the crystallite size of about 92 nm was formed. According to the obtained data, the adiabatic temperature was around 3282 K. This confirmed that the mode of the reaction was MSR.

![Fig. 8](image-url)  Morphological features of n-TiB₂ (a,b) before and (c) after the leaching process [46].
According to the SEM image in Fig. 8a, the morphology of the product before the leaching process was an agglomerate of oxide compounds (MgO and Mg₂TiO₄) and TiB₂. In this sample, the average agglomerate size was around 100 μm. Each agglomerate was composed of spheroidal fine particles with a mean size of about 129 nm as shown in Fig. 8b. From mechanochemistry point of view, when two adjacent primary particles collide, the coalescence may occur on the premise that these two particles share a common crystallographic orientation. Accordingly, two primary particles attach to each other and combine into a secondary one. Since the sizes of the secondary particles are still very small, it is reasonable that they will continue to collide and coalesce which may ultimately lead to the agglomeration [6]. From Fig. 8c, the product remained agglomerated even after the leaching process. However, the average agglomerate size of the leached sample declined to 500 nm. Moreover, the mean particle size of TiB₂ powder rose to 140 nm after the leaching process.

### 2.2.2 Mechanically alloyed nanocomposites in TiO₂–B₂O₃–Mg–C quaternary system

We recently studied the effect of the simultaneous presence of magnesium and graphite on mechanosynthesis of various nanocomposite powders in TiO₂–B₂O₃–Mg–C quaternary system [47]. To this end, a mixture of boron oxide and titanium dioxide powders along with different amounts of magnesium and graphite was milled using a high-energy planetary ball mill to provide necessary conditions for the occurrence of MSR. Results showed that T_{ad} was 3282 K in the absence of C (100 wt% Mg) and declines to 3012 K, when the graphite value increased to 20 wt%. The reduction in T_{ad} by increasing the C content was related to a decrease in the heat liberated per unit mass of reactant powders. In fact, graphite in the reaction acting as diluent and reducing the heat content which causes a reduction in T_{ad}. However, the values of T_{ad} were higher than 1800 K (in the absence and presence of 10 to 20 wt% C), thus the mode of the reaction was MSR. In the presence of x wt% Mg–y wt% C (x = 85 and 90; y = 100-x), the composite powders were composed of TiB₂ and TiC as major compounds as well as MgO and Mg₃B₂O₆ as unwanted phases. With further increasing the graphite content to 30 wt%, no activation was detected after 90 min of milling.

![Reaction Mechanism](image)

**Fig. 9** Reaction mechanism of the formation and SEM images of nanocomposite powders in TiO₂–B₂O₃–Mg–C quaternary system. (a) 90 wt.% Mg–10 wt.% C (S2), (b) 85 wt.% Mg–15 wt.% C (S3), (c) 80 wt.% Mg–20 wt.% C [47].

The reaction mechanism of the formation and SEM images of nanocomposite powders in TiO₂–B₂O₃–Mg–C quaternary system are shown in Fig. 9. According to this figure, it is evident that the formation of the nanocomposite powders in the above-mentioned system was strongly influenced by the type and weight fraction of the reductants. At
the beginning of milling, the system involved four ductile and soft powders. With continued deformation, the ductile system changed to ductile–brittle system by forming the brittle ceramic components. The brittle components such as Mg$_2$TiO$_4$ and MgB$_2$O$_4$ were dispersed in the ductile matrix that caused the work hardening and transformation of ductile particles into lamellar shapes. The brittle constituents were closely spaced along the interlamellar spacings. With further milling, the lamellae and brittle particles got further refined, the interlamellar spacing decreased, and the brittle components got uniformly dispersed. In fact, the equilibrium between fracturing brittle particles by trapping and ductile particles by work hardening got fine size, and made good dispersion in the matrix. From the SEM images, the morphology of the products exhibits agglomerates of unwanted phases (MgO and Mg$_3$B$_2$O$_6$) and desired constituents (TiB$_2$ and TiC). It is clear that the nanocomposite powders had a bimodal particle size distribution characterized by the presence of several coarse particles (≈250 nm) along with finer particles with the mean size of about 75 nm.

### 2.2.3 Mechanically alloyed nanocomposites in TiO$_2$–B$_2$O$_3$–Mg–Al quaternary system

In a similar system, we recently explored the mechanochemical behaviour of TiO$_2$–B$_2$O$_3$–Mg–C quaternary system [48]. In this regard, a mixture of B$_2$O$_3$ and TiO$_2$ powders along with various values of Mg and Al was milled using a high-energy planetary ball mill. Results showed that $T_{\text{ad}}$ was 3282 K in the absence of Al (100 wt% Mg) and declined to 3097 K, when the Al content increased to 100 wt%. Since the heat generated by the magnesiothermic reaction is higher than that by the aluminothermic reduction, $T_{\text{ad}}$ decreased quickly with increase of the Al content and reached a minimum in the presence of 100 wt% Al. However, the values of adiabatic temperature were higher than 1800 K and as a result the mode of the reaction was MSR. In the presence of 40 wt% Mg–60 wt% Al, the mechanochemical reaction was completed after 37–40 min which led to the formation of TiB$_2$–MgFe$_{0.6}$Al$_{1.4}$O$_4$ composites. In the case of 10 wt% Mg–90 wt% Al, a ternary nanocomposite (TiB$_2$–MgAl$_2$O$_4$–Al$_2$O$_3$) was formed after 43 min of milling. Besides, in the absence of Mg (100 wt% Al), Al$_2$O$_3$–TiB$_2$ nanocomposite was formed after 90 min of milling. Based on the obtained data, in the presence of 30 wt% Al, TiO$_2$ and B$_2$O$_3$ were initially reduced to Ti and B by aluminothermic and magnesiothermic reactions, respectively. Besides, the remaining TiO$_2$ was reduced by Mg. In the second step, the reaction of elemental Ti and B as well as MgO with Al$_2$O$_3$ led to the formation of TiB$_2$ and MgAl$_2$O$_4$, respectively. With increasing the Al content to 60 wt%, a similar trend was observed.

![Fig. 10](image_url)  
(a) EDS analysis and (b) elemental mapping images of the milled sample in the presence of 70 wt% Mg–30 wt% Al [48].

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According to the EDS spectrum (Fig. 10a), the main components of the composite nanopowder in terms of element were oxygen, magnesium, titanium, and aluminium. It should be noted that boron which was also present in the sample, could not be detected due to the strong absorption of the boron Kα line during the EDS analysis. Besides, Fe contamination caused by the excessive adhesion of powders to the vial and balls was not detected owing to the low relative Fe weight fraction in the milled sample. From elemental mapping analysis (Fig. 7b), the composite nanopowder had a homogenous distribution of O, Mg, Al, and Ti elements which confirmed the formation of a uniform microstructure after 37 min of milling. As shown in Fig. 11, the mechanosynthesized powders had high tendency to agglomerate. When the Al content reached 60 wt%, a unique flower-like structure was formed. From FE-SEM/FE-STEM images, it is obvious that the flower-like clusters were composed of loosely arranged nano-sheets with a side length of about 400 nm and a thickness of about 25 nm. It can also be seen that some nano-sheets become slightly curved which may result from internal stress or other factors [50].

3. Conclusions

In this review, a part of the recent developments of nanostructured materials from the mechanochemical processes have been reported. Results showed that a wide range of nanostructured materials can be produced by both progressive and mechanically induced self-sustaining reactions. In the progressive mode, the reaction may extend to a very small volume during each collision, resulting in a gradual transformation, while in the MSR manner a self-propagating combustion reaction can be initiated after a certain time. Based on the obtained data, the structural and morphological features of the mechanosynthesized nanopowders were influenced strongly by the processing parameters. From the microscopic observations, in many cases, the product had high tendency to agglomerate. However, each agglomerate was composed of fine particles (< 100 nm) with various morphologies. In a nutshell, the mechanochemical method not only is an interesting approach from theoretical point of view but also has many desirable features for industrial applications in which simplicity and high productivity are of primary importance. Nevertheless, the evaluation of the morphological characteristics of the milled powders is still a much-discussed question because no general shape factor available that clearly differentiates all possible kinds of shapes.

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