

Topics in Mining, Metallurgy and Materials Engineering
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Abdel Salam Hamdy Makhlouf
Gomaa A. M. Ali *Editors*

Waste Recycling Technologies for Nanomaterials Manufacturing

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Waste Recycling Technologies for Nanomaterials Manufacturing

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Preface

Nowadays, nanomaterials (NMs) are used in many areas and applications, including medicine, energy, and environment. The initial cost of the NMs is high; thus, finding cheap sources is required. In addition, waste accumulation is a serious environmental problem. Therefore, recycling waste into valuable NMs is highly required, where it has environmental and economic benefits.

Waste management is pressing hard to warn the industry. Humans always produce waste and discard it in some way, influencing the environment. At present, no spot on the earth is not exposed to some waste. These materials may cause immediate health risks to humans and animals. Other wastes persist for a long time in the environment until they reach damaging levels to ecosystems. Hence, the upsurge in waste generated by the industries and human activities needs to be managed. Various recycling methods have been developed and applied for the conversion of wastes into useful forms of materials and NMs. The standard methods applied to recover the generated wastes, including recycling, reducing, and reuse, still need more developments.

Information and techniques for investigations are minimal. Nonetheless, it is incredibly likely that NMs used in several items would be in the waste stream. Environmental risks related to the treatment of nanowastes remain unexplored. Another factor is whether items containing NMs, consisting of recycling processes, will affect the waste management capabilities/performance or not. In comparison, NMs may substitute certain substances that make products, e.g., smarter or more efficient, to get into waste management sooner and potentially play a role in waste reduction. Draw up an overview of nanomaterial and waste-related scientific, health, and environmental problems, and assess the available recycling issues are needed. The ultimate goal is to consider looking for identical statistics to compare the potential hazards associated with the existence of NMs in the waste.

This book provides in-depth studies about these challenges and covers these issues in four parts.

Part One: Fundamentals, Current Prospects, and Future Trends

In this part, we covered the basics of nanomaterials in terms of manufacturing, characteristics, and applications. Various techniques used to recycle waste have been discussed. In addition, this part highlights the fundamentals, current prospects, and future trends of the recovered nanomaterials.

Part Two: Electronics Waste Recycling Technologies

In this part, we highlighted the importance of recycling in terms of environmental and economic perspectives. We discussed the recycling techniques of electronic waste, including lithium-ion batteries, zinc-carbon batteries, etc. For example, hierarchical cobalt oxide nanostructure has been recovered from spent lithium-ion batteries using magnetic electrodeposition. In addition, MnO_2 nanoflower has obtained from zinc-carbon batteries using electrodeposition and other methods. The materials used for manufacturing lithium-ion batteries also recovered from various waste sources. The applications of the recovered materials for supercapacitors, batteries, electrocatalytic, and sensing have been discussed.

Part Three: Agriculture Waste Recycling Technologies

In this part, we covered the conversion of agricultural waste into nanomaterials, mainly carbon-based nanomaterials and their composites. The studied agriculture waste includes rice husk, rice husk ash, bamboo leaves, bio-waste sago bark, banana fibers, argania spinosa seeds, corn grains, sugarcane fibers, and oil palm shells, palm kernel shells, orange peel, wheat flour, etc. Various nanomaterials compositions and morphologies were obtained, such as pure activated carbon, hetero-atom-doped carbon materials, and metal oxides/carbon nanocomposites. The recovered materials have been studied for various applications, including water treatments, energy storage, and forensic medicine applications.

Part Four: Plastic and Polymeric Waste Recycling Technologies

Plastic is one of the most significant hazards to the environment. Plastic is a non-biodegradable material, and several toxic chemicals leach out of it and seep through the soil, water, plants, and animals. In this part, we introduced the topic of utilizing plastic wastes as a precursor for the fabrication of carbon-based materials. While also highlighting the factors affecting the efficiency of each process and the recent progress in this regard, this part also highlights recycling polyethylene terephthalate waste into a novel magnetic nanoadsorbent. Recent breakthroughs in

carbon-based nanomaterials' science and technology use paraffinic waxes as a carbon source where it consists of not less than 18 carbon number per single paraffin crystal. This part also describes the separation of paraffinic petroleum wax, its purification, and characterization beside nanocarbon synthesis. Different nanomaterials can be synthesized from the waste plastics, such as polyvinyl chloride plastic is used as the carbon source for the fabrication of MoC_2 nanoparticles.

Cairo, Egypt
Edinburg, USA
Assiut, Egypt
November 2020

Abdel Salam Hamdy Makhlouf

Gomaa A. M. Ali

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Dr. Makhoulf is the author of over 200 peer-reviewed journal and conference papers, 19 books and handbooks, 30 book-chapters, as well as +100 technical reports. The h-index is 37, with > 4570 citations. Many of his publications have been ranked among the World's Best in the fields of Nanostructures, Nanomaterials, Biomedical Engineering, Materials Science, Coatings, Environmental Science, Nuclear Materials.



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Fundamentals, Current Prospects, and Future Trends

Fundamentals of Waste Recycling for Nanomaterial Manufacturing



Gomaa A. M. Ali and Abdel Salam Hamdy Makhlouf

Abstract Nowadays, nanomaterials are used in many areas and applications, including medicine, energy, and environment. The initial cost of the nanomaterials is high; thus, finding another cheap source is required. In addition, waste accumulation is a serious environmental problem. Therefore, recycling waste into valuable nanomaterials is highly required, where it has environmental and economic benefits.

Keywords Nanomaterials • Waste • Recycling • Nanotechnology • Manufacturing

List of Abbreviations

CVD	Chemical vapour deposition
mZVI	Meso-scale zero valent iron
NSMs	Nanostructured materials
PVD	Physical vapour deposition
ppb	Parts per billion
SG	Sol–Gel
TEOS	Tetraethylorthosilicate

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1 Fundamentals of Nanomaterials Manufacturing

1.1 Nanoscience and Nanotechnology

Nanoscience is one of the hottest areas and rapidly expanding research fields. It aims to synthesize, study, characterize, and evaluate new materials, for which one or more of their dimensions lies in the nanometer scale (1–100 nm). Nano refers to the 10^{-9} power or one billionth. Nanotechnology aims to fabricate these materials in the form of devices systems and other industrial products. Advances in nanoscience lead to new developments in nanotechnology. Materials of this size are particularly attractive because of their inherent physical and chemical characteristics because of their high surface-to-volume ratio. Nanoparticles are very sensitive to surface environments. Nanotechnology aims to better understanding these properties and to find new ways of utilizing them in our daily life.

Nanomaterials have received a great deal of research interest because of their unique catalytic [1, 2], optical [3, 4], electrical [5–7], magnetic [8, 9], sensing [10–12], storage [13–16], and mechanical and chemical properties. Nanomaterials have unique characteristics than when in bulk (micro or macro) form. Nanomaterials can be classified into several categories based on their structures such as nanoparticles, nanotubes, nanoflowers, nanocapsules, nanocoatings, nano-thin-films, nanorods, nanoflakes, nanofibers, nanocarriers, nanoceramics [13, 17–25], and nanocomposites. The nanomaterials' applications can also be classified as nanomagnetism, nanomedicine, nanotoxicology, nanoelectrochemistry, nanoengineering, nanoscience, etc. [26–29].

1.2 Types of Nanomaterials

Solid substances can be divided into metals, ceramics, semiconductors, composites, and polymers [6, 7, 10, 30–36]. When the size of these materials is decreased to the nano-range, they can be further subdivided into nanoparticles, nanocrystals, nanotubes, nanorods, and nanocomposites [14, 15, 37–40]. The first classification scheme of nanostructured materials (NSMs) was provided by Gleiter in 2000 [41], and other classification was extended by Pokropivny and Skorokhod [42] as shown in Fig. 1. 0D, 1D, 2D, and 3D are the main classifications of NSs based on their dimensionality.

1.3 Nanosized Structures

Nanomaterials are the bridge between bulk materials and atomic structures. The physical properties of the bulk materials are constant regardless of its size, but for

Dimensionality classification of nanostructures ($L < 100 - 500 \text{ nm}$)

Designation: dimensionality of NS $\rightarrow$ $kDlm.n$ $\leftarrow$ dimensionality of elementary units
 $k \geq 1, m, n \quad \{k, l, m, n\} = \{0, 1, 2, 3\}$

Elementary building units :

1. 0D  Molecules, clusters, fullerenes, rings, metcarbs, thoroids, domens, particles, powders, grains, schwartzons	2. 1D  nanotubes, fibers, filaments, whiskers, spirals, belts, springs, horns, columns, needles, pillars, helicoids, wires, ribbons	3. 2D  layers
--	---	---

0D-nanostructures :

4. 0D0  uniform particles arrays	5. 0D00 hetero-geneous particles arrays, "core-shell" dendrimers, onions 
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1D-nanostructures :

6. 1D0  molecular chains, polymers	7. 1D00  heteropolymers	8. 1D1  bundles, ropes, cables, corals	9. 1D11  heterochains, heterocables, saws, hair, heterobundles, junctions, combs, bows	10. 1D10  beads, pea-pods, fullereno-fibers
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2D-nanostructures :

			
fullerene films		nanostraw, PhC, fibers films	
13. 2D2 tiling, mosaic, layered films 	14. 2D00 heterofilms of heteroparticles, fullereno-powders 	15. 2D10 films of pods, fullereno-fibers 	16. 2D11 films of fibers and nanotubes, PhC-waveguides 
17. 2D20 fullereno-plate films 	18. 2D21 bridges, fiber-layer films 	19. 2D22 hetero-layers, MOS-structures 	20. 2D210 fullerene-fiber-layer films 

3D-nanostructures :

21. 3D0  Fullerites, clathrates, powder skeletons, log	22. 3D1  skeletons of fibers, nanotubes	23. 3D2  layer skeletons, buildings, honeycombs, foams	24. 3D00  sols, colloids, smogs, heteroparticles composites
25. 3D10  skeletons of fibers-powders	26. 3D11  skeletons of heterofibers, nanotubes	27. 3D20  intercalates, skeletons of layers and powders	28. 3D21  Cross-bar-layers, layer-fiber skeletons
29. 3D22  heterolayers	30. 3D30  opals, dispersions, particles, pores, fullerenes in matrix	31. 3D31  membranes, PhC, fiber composites, waveguides	32. 3D32  friction pairs, contacts, interfaces, cavities, grain boundaries
33. 3D210  composites of layers, fibers and particles in matrix	34. 3D310  membranes + impurities, powder-fiber composites	35. 3D320  powder-layers composites	36. 3D321  layers-fibers-composites in matrix, VCSEL

Notices :

1. Interfaces between building units not regarded as additional 2D-NSs
2. Inverse NSs with cavity building units not regarded as separate ones
3. The classification may be extended with account of fourfold combinations

Fig. 1 Dimensionality classifications of nanostructured materials Adapted with permission from Ref. [42], Copyright 2007, Elsevier

the nanomaterials, the properties are varied with the variation in the particles size. Size-dependent properties are detected, such as quantum confinement [43] in semiconductor nanoparticles, surface plasmon resonance [44] in some metal nanoparticles, which are the fundamentals behind many colour-based biosensor applications and superparamagnetism [45] in magnetic nanomaterials. Nanocomposite materials can be considered as multiphase materials in which one or more from its components have nanometer dimensions [46]. One of its components is considering a guest phase, and the other is considered as the host one. The host phase acts as a matrix in which the guest phase is distributed [35], or as a shell coated the guest core [47, 48]. The nanocomposite materials exhibit properties of both the guest and host compounds [31, 49, 50].

2 Synthesis of Nanomaterials

The top-down and bottom-up are main approaches to nanomaterials synthesis. A typical top-down method is attrition or milling, whereas the bottom-up approach examples include colloidal dispersion and deposition. Synthesis routes play a crucial role in the properties of the target product. Many synthesis techniques have been evaluated and developed to obtain nanoscale materials with proper morphologies.

2.1 Vapor State Processing Routes

2.1.1 Physical Vapor Deposition

Thin-film nanomaterials could be easily prepared by physical vapor deposition (PVD). The process includes the generation of vapor phase species via sputtering [51], laser ablation [52], or plasma spray [53] (Fig. 2), and then the vapor is condensed onto a substrate followed by the nucleation and growth. The process has some limitations in the case of preparation of the multicomponent materials (nanocomposites) due to the differences in the evaporation temperature of the components because of the differences in vapor pressures of the evaporating species.

2.1.2 Chemical Vapor Deposition

The process in which the gaseous species decompose or react on a heated surface to make stable solid products is called chemical vapor deposition (CVD) [55]. Metallic, ceramic, and semiconducting thin films could be deposited using CVD. The process can be classified into thermally activated, laser-assisted, and

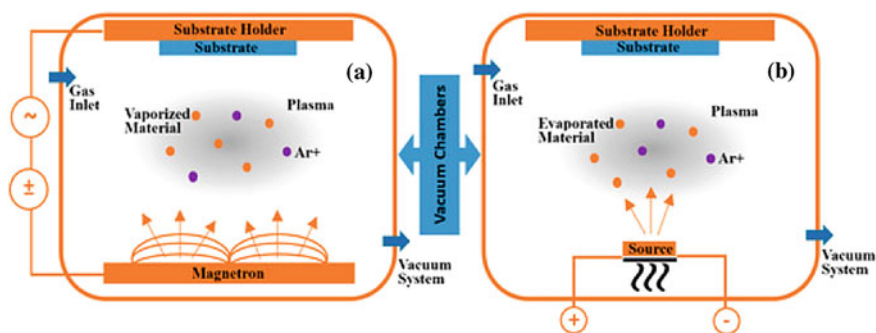


Fig. 2 Schematic illustration of conventional PVD processes (sputtering (a) and evaporating (b)). Adapted with permission from Ref. [54], Copyright 2018, MDPI

plasma-assisted CVD based on the activation sources for the chemical reactions [56]. Figure 3 shows the setup of a horizontal CVD and parallel-plate plasma-enhanced CVD reactors. In the case of metal chloride precursors, corrosive chlorine-containing by-products are formed [56]. CVD is a more complex method of forming thin films and coatings than PVD due to the surface and gas phase interactions.

2.1.3 Spray Conversion Processing

The chemical precursors could be atomized into aerosol droplets that are dispersed throughout a gas atmosphere, then moved into a heated reactor to form thin films or ultrafine particles. The atomization purposes use various aerosol generators, including pressure, electrostatic, or ultrasonic atomizer. Spray pyrolysis is the most commonly used aerosol processing method [58].

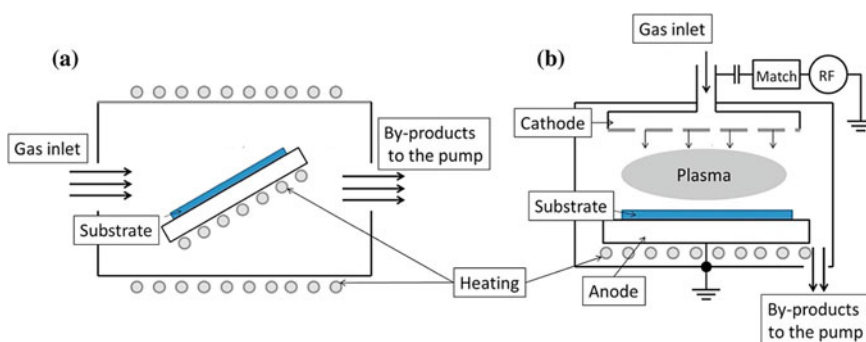


Fig. 3 Setup of a horizontal CVD (a) and a parallel-plate plasma-enhanced CVD (b) reactors. Adapted with permission from Ref. [57], Copyright 2019, Elsevier

2.2 *Liquid State Processing Routes*

2.2.1 Sol–Gel Method

The sol–gel (SG) route has been used for metal oxide and ceramic powders production with high homogeneity and purity [10, 35, 59, 60]. SG process involves the formation of a colloidal suspension (sol), which is converted to viscous gel and solid material. In the process, reactive metal precursors were hydrolyzed, followed by condensation and polymerization reactions. Many types of metal precursors can be used (alkoxides, carboxylic salts, chlorides, nitrates, etc. [61]). To achieve gel densification, the solvent removal and appropriate drying are required. Tetraethylorthosilicate (TEOS; $\text{Si}(\text{OC}_2\text{H}_5)_4$) is the most common studied metal alkoxides [62]. However, the conversion of metal precursor molecules needs acid or base catalysts such as sulphuric acid or ammonium hydroxide since the hydrolysis of silicon alkoxides is very slow. The conversion from a solution containing individual solvated particles to a colloidal three-dimensional network is represented in Fig. 4a. In addition, Fig. 4b, c shows the detailed synthesis process of $\text{CoO}_x\text{--SiO}_2$ system. The most advantages of the SG method are high purity, good homogeneity of the prepared materials, lower preparation temperature, precise composition control, versatile shaping, and preparation by cheap and straightforward apparatus compared with other methods.

2.2.2 Citrate-Gel-Pechini Process

In the citrate-gel-Pechini method, an organic network formed in precursor solutions; thus, fine oxide powders are obtained after a heating process stabilizes metal ions. This method can be used for the preparation of multicomponent compositions with good homogeneity and control of stoichiometry [64]. The citric acid is used as a capping agent because of its relatively strong multifunctional organic acid. Pechini route utilizes poly-chelates between the metal ions and C=O ligands of citric acid [65]. The chelating process takes place during the aggressive stirring of precursor solution containing metallic salts and CA. Figure 5 shows Pechini process for CuO/CeO_2 nanocomposites preparation. The simplicity of the method, homogeneous microstructure, and low crystallization temperature of the obtained material are the main advantages of the Pechini route [64]. Different types of precursors can be used. Moreover, both thin films and nanocomposite powders can be obtained [64, 66]. Higher surface area and lower particle size products can be obtained.

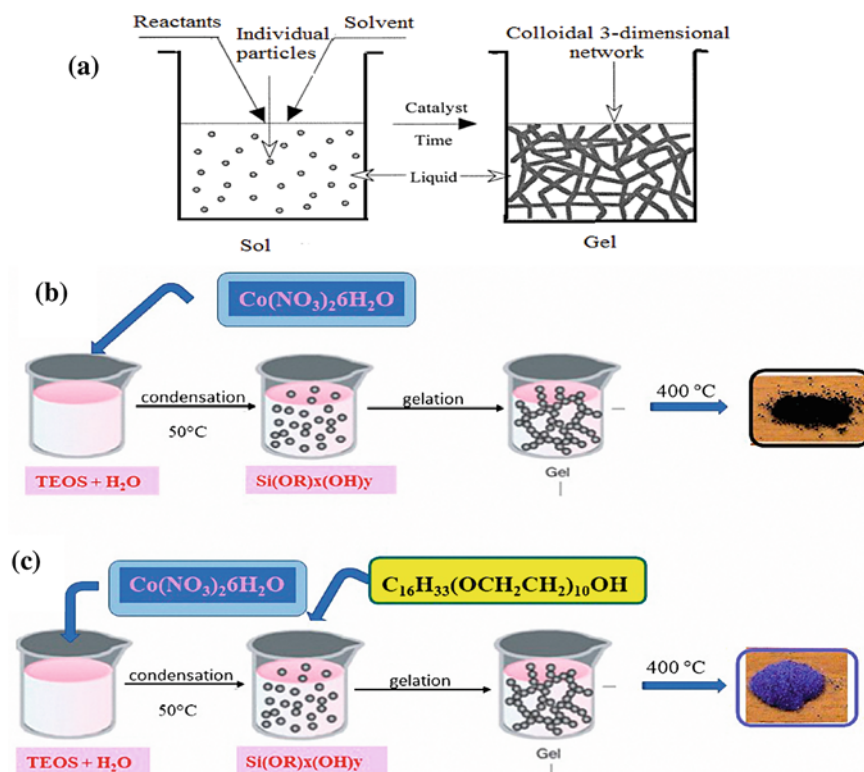


Fig. 4 Simple representation for sol-to-gel transformation (a) sol-gel process of $\text{CoO}_x\text{-SiO}_2$ preparation in presence of (b) hydrolytic alkoxide and non-ionic surfactant (c). Adapted with permission from Ref. [63], Copyright 2019, MPDI

2.2.3 Wet Chemical Synthesis

This simple and single-step method includes chemical reaction producing different shapes nanorods [68], and nanoparticles with approximate spherical shape [69]. Various nanostructured forms (nanoflowers, nanoneedles, and staking of flake-like structures) are achieved by controlling the experimental conditions [70]. Both organic and inorganic additives can yield different shapes of nanomaterials products. The shape of particles can be controlled by controlling the adsorption of some inorganic anions to particular crystal faces [71]. ZnO nanopencils have been synthesized using wet chemical method as shown in Fig. 6 together with its morphological investigations.

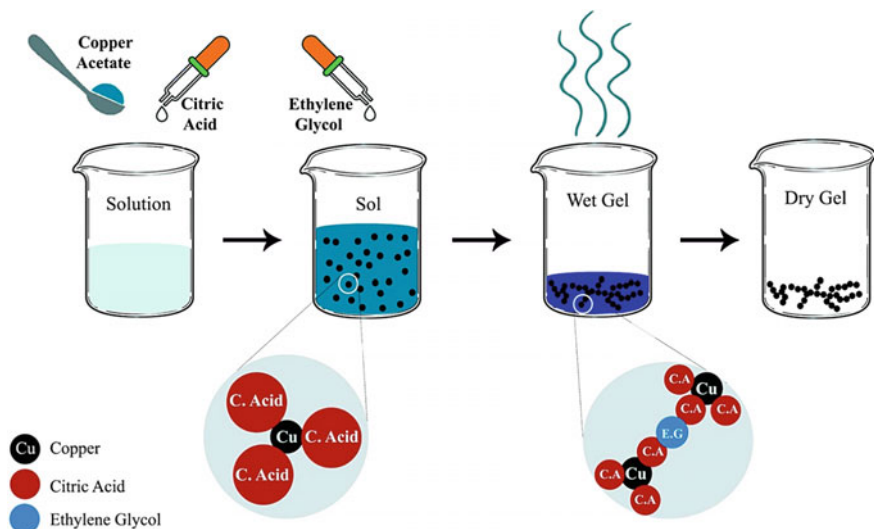


Fig. 5 Pechini process for CuO/CeO₂ nanocomposites preparation. Adapted with permission from Ref. [67], Copyright 2019, Elsevier

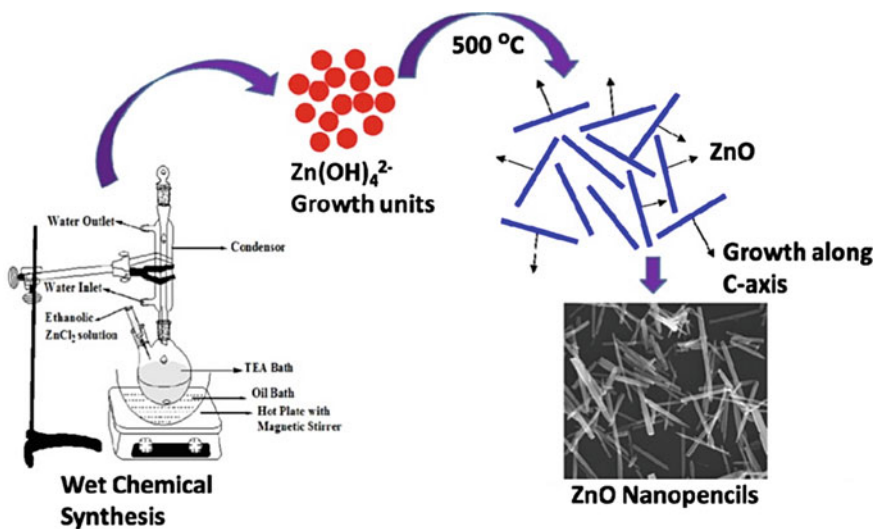


Fig. 6 Synthesis of ZnO nanopencils using wet chemical method. Adapted with permission from Ref. [72], Copyright 2016, Elsevier

2.3 Solid-State Processing Routes

2.3.1 Mechanical Milling

The particles' size of metal and ceramic materials can be reduced to the nanoscale by high-energy ball milling. Some factors must be taken in the account such as: (i) the powder-to-ball mass ratio, and (ii) addition of process control agent [73]. The ball milling techniques can successfully reduce the particle size to few nanometers, but phase transformation may occur [73, 74]. Present size reduction methods possess some disadvantages, such as contaminations from grinding media. Figure 7 shows the formation mechanism of meso-scale zero valent iron (mZVI) nanoparticles as an example by high-energy ball milling process.

2.3.2 Mechanochemical Preparation

The mechanochemical preparation process of the nanomaterials includes mechanical activation of solid-state displacement reactions in a ball mill. Thus, chemical

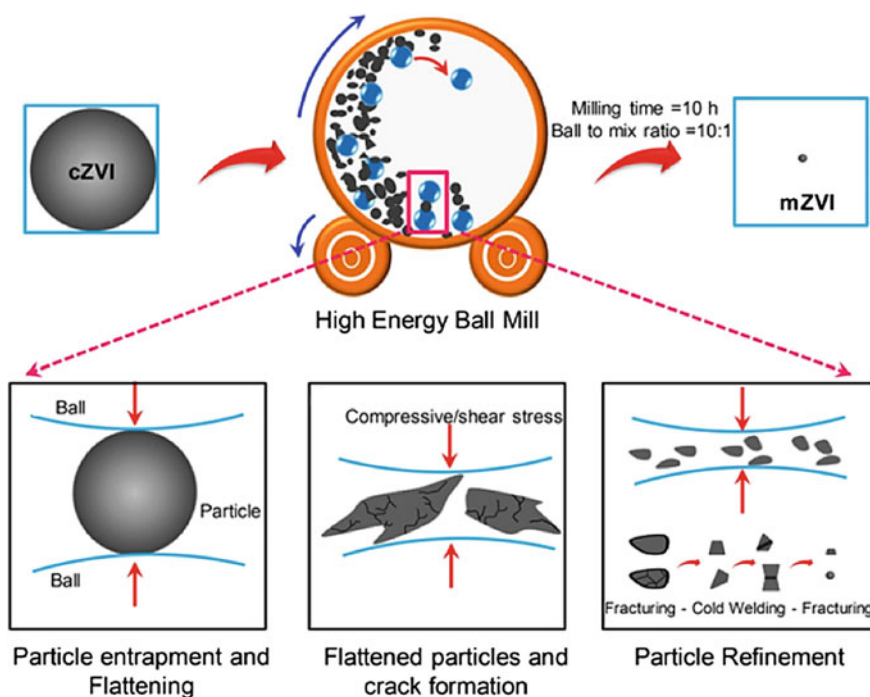


Fig. 7 Formation mechanism of meso-scale zero valent iron nanoparticles by high-energy ball milling process. Adapted with permission from Ref. [75], Copyright 2016, Elsevier